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DEVELOPMENT AND EVALUATION OF PG/SIC CODEPOSITED COATINGS FOR ROCKET NOZZLE INSERTS. VOLUME I. THERMAL AND MECHANICAL PROPERTIES OF PYROLYTIC GRAPHITE/SILICON CARBIDE CODEPOSIT

Richard H. Singleton

Atlantic Research Corporation

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The room temperature tensile strength was much greater than PG in the c direction, as expected as a result of the SiC crystal reinforcement. Tensile strength and elastic modulus were also greater than PG in the ab

direction to up 4000°F. The data must be considered to be preliminary and nonstatistical.

ABSTRACT

This three-volume document describes the fabrication development and evaluation of codeposited pyrolytic graphite-silicon carbide coatings on graphite substrates for a rocket nozzle application. The general purposes of the program were to establish a reproducible coating technique and to evaluate the coating by determination of its thermal and mechanical properties and by 1.0-inch throat diameter subscale rocket motor test firings. The study was preliminary to the fabrication and testing of 1.7-inch throat diameter PG/SiC lined nozzles in the High Pressure Solid Propellant Test Facility in the Rocket Propulsion Laboratory at Edwards Air Force Base. The work was done under the overall technical cognizance of the RPL Project Engineer, Mr. William F. Payne. The results obtained in these studies are presented in three separately bound volumes as follows:

Volume I – Thermal and Mechanical Properties of PG/SiC Codeposit

By determination of its thermal and mechanical properties, it was found that the PG/SiC coating is unique in that it is nearly isotropic with respect to thermal expansion and elastic modulus but quite anisotropic with respect to thermal conductivity. The tensile strength was much greater than pyrographite in the c direction, as expected as a result of the SiC crystal reinforcement. Mechanical properties in the ab direction were equal to or better than pyrographite.

Volume II — Vapor Deposition of Pyrolytic Graphite/Silicon Carbide Codeposited Coatings

Parameters were established to vapor deposit PG/SiC coatings on components for nozzles with throat diameters up to 1.7 inches.

Volume III – Subscale Solid Rocket Motor Testing of PO/SiC Lined Throat Inserts

Six ATJ graphite 1.0-inch throat diameter nozzle throat inserts and four entrance approach sections were coated with PG/SiC and test fired with a low oxidizing 21 percent aluminum 5600°F propellant. A preliminary throat erosion rate for PG/SiC of 4 mils per second at 2000 psia and 7 mils per second at 2500 psia average chamber pressure was established. Use of a graded coating in which the last third of the deposit was pure pyrographite approximately halved the erosion rate. The inserts appeared to be less prone to cracking when the ATJ substrates had been heat treated at 5400°F prior to coating.

The Tables of Contents for Volumes II and III are included here for completeness and cross-reference.

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SECTION I

INTRODUCTION

It was known that pyrolytic graphite (PG)/silicon carbide (SiC) was less anisotropic than PG and that this was due to the fact that the SiC reinforcing needles were perpendicular to the PG plane in the microstructure. Thus, the coefficient of thermal expansion (CTE) was decreased in the c direction, and the tensile strength was increased in the c direction. The present work was undertaken to measure the thermal (CTE, thermal conductivity) and mechanical properties (in both tension and compression) up to 4500 or 5000°F. The objective was to procure engineering data for use in structural analysis for the design of the 1.7-inch throat diameter HIPPO nozzle.

Although (a) a separate individual run had to be made to fabricate each specimen, (b) only single point data was obtained in most cases, and (c) process control (especially SiC content) was less than desired, it is felt that a good set of preliminary data was procured.

At this writing, new data has been obtained under the SCALE UP program on the CTE of PG/SiC in the ab direction which is 10 to 20 percent higher than the data reported in this report. Since CTE is the most important single property with respect to structural analysis, the reader should be cautioned not to use this CTE data before consulting with Atlantic Research Corporation.

SECTION II

OBJECTIVE

Procure a preliminary nonstatistical data package on CTE and thermal conductivity, and generate stress-strain curves in both the compressive and tensile modes, as functions of material orientation; i.e., a and c direction.

SECTION III

DATA HIGHLIGHTS

The work that was completed at Southern Research Institute (SoRI) is summarized in Table I.

1. THERMAL PROPERTIES

The CTE was determined over the temperature range 70 to 5000°F in both the a and c directions for PG/SiC compositions ranging, nominally, from 0 to 25 weight percent SiC. The thermal expansion data is

Table I. Test Matrix for PG and PG/SiC Properties by SoRI.

	2	8% SiC	15% SiC	20% SiC	25% SiC	PG STOCK	PG/Sic STOCK
THERMAL CONDUCTIVITY							
ab DIRECTION < 1500°F	7	ı	i	2	2	PLATE	TUBE AND PLATE
c DIRECTION < 1500°F	7	1	ı	2	2	PLATE	TUBE AND PLATE
c DIRECTION > 2000°F	1	ı	;	m	۳	ı	TUBE
THERMA! EXPANSIGM (70 to	in SOON'E)	ũ					
ab DIRECTION	8	8	7	2	7	TUBE	TUBE AND PLATE
c DIRECTION	7	2	7	2	7	PLATE	TUBE AND PLATE
TENSION (σ/ϵ)							
ab DIRECTION 70°F	-	ı	7	2	-	PLATE	TUBE
ab DIRECTION 3000°	t	ı	2	2	-	I	TUBE
ab DIRECTION 4000°F	ı	1	7	2	_	ı	TUBE
ab DIRECTION 4500°F	-	ı	7	e	7	PLATE	TUBE
C DIRECTION 70°F	٣	1	က	ı	4	TUBE	TUBE
COMPRESSION (0/e)							
ab DIRECTION 70°F	2	ı	S	7	7	TUBE	TUBE
ab DIRECTION 3000°F	1	ı	_	i	7	ı	TUBE
ab DIRECTION 4500°F	t	ŧ	2	ı	-	ì	TUBE
c PIRECTION 70°F	1	1	1	m	ı	ı	TUBE

NOTE: NUMBERS REPRESENT NUMBER OF TEST REPLICATES

summarized in Figure 1. It has recently been discovered that the SiC contents are higher than formerly supposed and Figure 1 contains the corrected compositions. It may be observed in Figure 1 that this first cycle data shows that the addition of 18 percent or more SiC to PG renders it nearly isotropic. It also appears improve the thermal stability of the PG, since the rapid rise in the ab direction PG curve above 4000° F is eliminated. These CTE data are preliminary and should not be used for engineering purposes without consultation with Atlantic Research. The data is plotted on smaller coordinates in the Southern Research Institute Report 73-031 which is attached to this report as Appendix I. For comparative purposes, the Δ/L of the ATJ graphite coating substrate has recently been determined to be 1.01 percent in the with grain direction, between 70 and 4000° F. A good match between the CTE of ATJ and that of PG/SiC in the ab direction is evident.

The coatings were made on graphite substrates in either 2-inch-diameter cylindrical or plate form using a thin PG layer predeposit to facilitate separation from the substrate. Pure PG was laid down at 4000°F on AGSR graphite; whereas, PG/SiC was deposited at 3200°F on ATJ graphite. The microstructures consisted of fine acicular fairly well dispersed particles generally aligned perpendicular to the substrate surface and 0.5 to 1.0 micron in thickness with an L/D ratio greater than 3. The coatings were made during the spring of 1972.

Specimens 3 inches long were machined with their lengths in the coating gas flow direction from plate, or cylinders, for the ab plane CTE measurements. The c direction CTE specimens were fabricated by cutting disks from plate, or similar round shapes from cylinders, and stacking them to form a pile 1 inch high. All measurements were made at SoRI using a graphite dilatometer with a dial gauge.

The thermal conductivity of PG was determined between room temperature and 1700°F in both the ab plane and the c direction. The thermal conductivities of PG/20% SiC and PG/35% SiC were determined to 1700°F in the ab plane and to 4300°F in the c direction. The data are summarized in Figure 2, along with similar data for the ATJ graphite substrate taken from the literature. It may be observed that, although the addition of SiC to PG lowers the anisotropy to a considerable extent, PG/SiC is still quite anisotropic with respect to thermal conductivity. The data is plotted on smaller coordinates in Appendix I.

The ab plane and c direction comparative rod thermal conductivity specimens for measurement from room temperature to 1500° F were made from either plate or cylindrical stock. The ab specimens were fabricated by fitting accurately machined layers together to form a cylinder, 0.6 to 1.0 inch long by 0.75 inch in diameter, with the c axis of the material perpendicular to the specimen axis. The c direction comparative rod specimen was a single layer flat disk or an arc-disk cut from a cylinder. The method utilizes a common heat flux flowing in series through the unknown specimen and one or more specimens of known thermal conductivity. Measurement of the ΔT through each unit allows a simple calculation of the unknown thermal conductivity. Above 1500° F, excessive heat losses make this method inaccurate and the radial in-flow method is used.

In their radial heat flow method, a known heat flux was passed through the PG/SiC cylindrical specimen in the c direction and the ΔT across the specimen was measured by optical pyrometry. The heat flux was measured by water flow calorimetry. Measurements were made from 2300 to 4300°F. The data appeared to blend well with the comparative rod data. The PG/SiC specimens were 2 inches in O.D. \times 2 inches long by approximately 150 mils thick.

¹See Figures 18 and 20 of Appendix A.

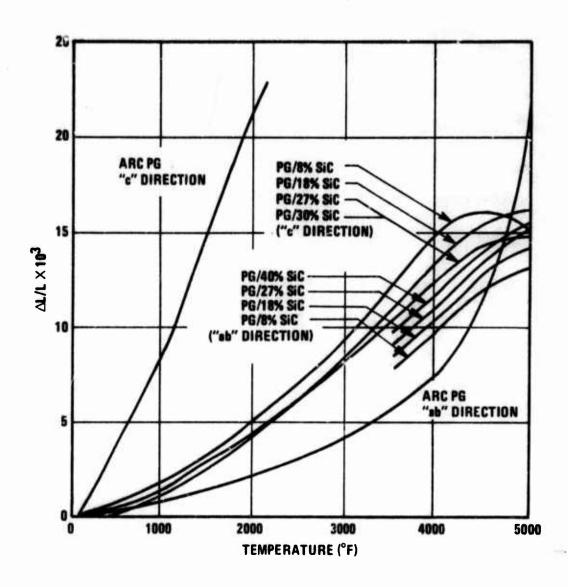
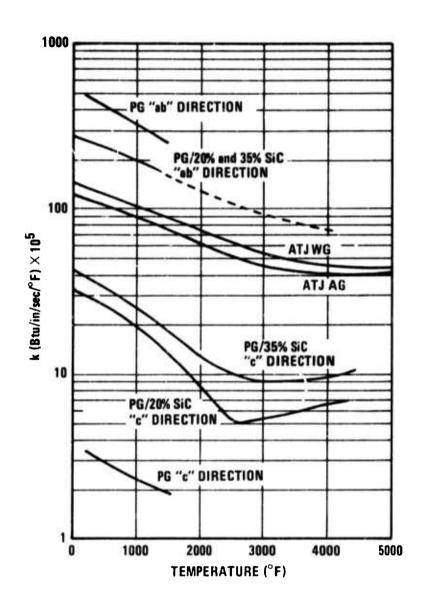


Figure 1. Thermal Expansion of PG and PG/SiC Codeposit.



References								
PG	ab	Appendix B	Figure 15					
PG	C	Appendix B	Figure 16					
PC/20% SiC	ab	Appendix B	Figure 17					
PG/20% SiC	C	Appendix B	Figure 18					
PG/35% SiC	ab	Appendix B	Figure 19					
PG/35% SiC	C	Appendix B	Figure 20					

ATJ WG&AG Private communication 1972 from SRI

Figure 2. Thermal Conductivity of PG and PG/SiC Codeposit.

During the coating material fabrication for thermal conductivity specimens, the pure PG was deposited at 4000°F, to a depth of 90 to 200 mils; whereas the PG/SiC was deposited at 3200°F to a depth of 110 to 180 mils. The microstructure of the PG/SiC generally consisted of fine acicular fairly well dispersed particles aligned perpendicular to the substrate surface and with an L/D ratio greater than 3. The thermal conductivity coatings were made in May through August 1972, except that the PG plate was fabricated in October 1972.

2. MECHANICAL PROPERTIES

Two-inch-I.D. by 9-inch-long graphite cylinders coated on their inside diameters with PG/SiC to a depth of 100 to 150 mils in the gauge section were submitted to SoRI for tensile meeasurements. Similar tubes 5 inches long were submitted for compression testing. This latter shipment included some pine PG 40 to 60 miles thick. Some PG plate 50 mils thick was also submitted for tensile testing. The PG was laid down at 4000°F on AGSR cylinders and the PG/SiC was deposited at 3200°F on ATJ graphite cylinders. The microstructure of the PG/SiC generally consisted of fine, acciular, well dispersed SiC in a crack-free PG matrix. The coatings were made during the summer of 1972. The SiC content was determined at SoRI by ashing of material taken from the gauge area of broken room temperature specimens. Compositions varied considerably from nominal and, consequently, data was obtained at more compositions than originally planned. Although the number of replicates and temperatures were small, some fairly definite trends were established.

The results showed that the addition of SiC to PG increased the tensile strength almost linearly over the temperature range 70 to 3000°F. Above 3000°F, the strengthening effect of the SiC began to decrease and was significantly diminished, but still present, at 4500°F. The tensile elastic modulus and strain-to-failure both increased with SiC content over the entire temperature range, 70 to 4500°F. The compressive glastic modulus similarly increased with SiC content up to 3000°F, but the effect was much less pronounced at 450.0°F.

The complete mechanical properties data and techniques used in their determination at SoR1 are contained in their Report SoR1-EAS-73-183 which is attached to this report as Appendix II.

a. Tensile Properties

Typical ab plane stress-strain curves in tension are shown in Figures 13 and 14 of Appendix II for PG/16% SiC and PG/20% SiC, respectively, as a function of temperature. It may be observed that the curves show a nearly linear elastic bahavior up to and including 3000°F; whereas above this temperature the curves are bilinear showing some plastic deformation. The ultimate tensile strengths are plotted as functions of SiC content and temperature in Figure 3. It may be seen that the strength peaks at approximately 3000°F and then drops, at 4500°F, to near the room temperature values for the lower SiC contents and to below the room temperature values for the high SiC contents. The tensile modulus of elasticity is plotted as a function of SiC content and of temperature in Figure 4. Again, the modulus values peak at near 3000°F.

The original ab plane specimens were fabricated at SoRI by machining away the substrate and some of the coating in the gauge section. However, breakage occurred in room temperature testing in the specimen heads prior to breakage in the gauge section. Consequently, arc-of-cylinder specimens were fabricated with conforming grips. These were used successfully throughout the program, and stress-strain curves made with full cylinders coincided fairly well with these arc coupons at room temperature. The PG coupon specimens were made from plate.

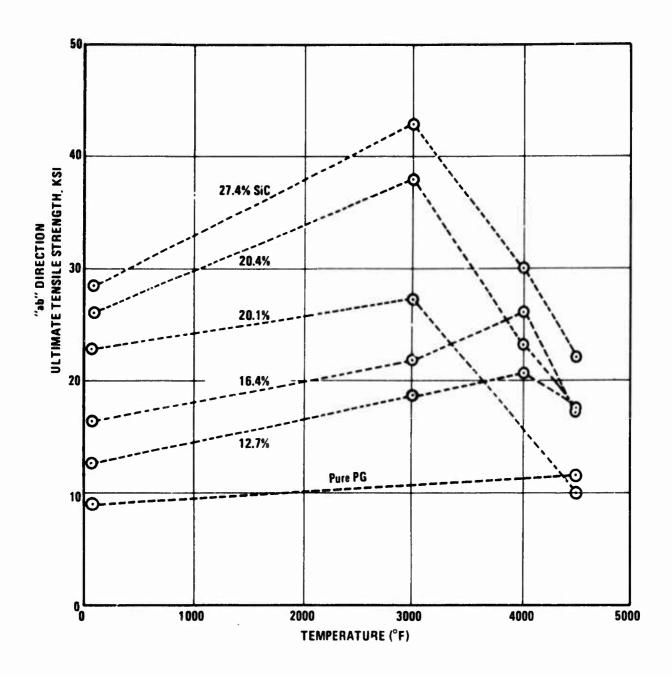


Figure 3. Strengths of PG/3:C Codeposit in the "ab" Plane.

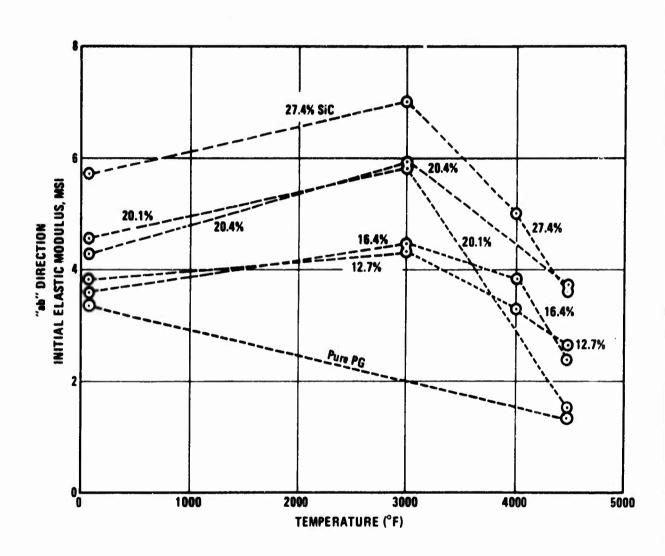


Figure 4. Moduli of Elasticity in Tension of PG/SiC Codeposit in the "ab" Plane.

For the c direction tensile tests, conforming grips were epoxy bonded to arc-disk specimens cut from cylinders. The pure Atlantic Research PG tensile strength averaged 792 psi; whereas, the epoxy bond broke at 3,000+ psi in the case of the PG/SiC specimens at room temperature. Previous privately communicated measurements in the c direction at SoRI had indicated strengths near 8,000 psi for PG/SiC at room temperature. Insufficient length was available in the c direction for the use of strain gauges so that the elastic modulus and stress-strain curves were not obtained.

Poisson's ratio in tension was determined to be 0.14 for PG/20% SiC at room temperature in the a/b mode.

b. Compressive Properties

The compressive strength specimens were fabricated by machining off the graphite substrate and some of the coating in the gauge section, using Atlantic Research supplied cylinders shorter than those used for tensile testing. In addition, are coupon specimens were fabricated similar to but shorter than those used for tensile testing. The coupon specimens were used at temperature. Room temperature testing with are and full cylinder specimens indicated good agreement in data. The specimens buckled in testing above a certain strain level. SoRI did not report the data beyond this point and consequently ultimate strength and strain-to-failure were not obtained. However, SoRI claims that the stress-strain curves are accurate up to the point of buckling.

The compressive stress-strain curves are fairly similar to the tensile curves for the first 0.5 percent strain. However, they are bilinear at all temperatures. As can be seen in Figure 16 of Appendix II, the elastic modulus in compression is quite similar to, although slightly less than, that in tension, peaking at approximately 3000°F.

Room temperature stress-strain curves were obtained in compression in the c direction on 160-mil-thick disks taken from Atlantic Research plate Run 001-47. This PG/SiC material contained approximately 20 percent SiC. The stress-strain curves are shown in Figure 5. The data was not reported in Appendix II.

Poisson's ratio in compression was determined to be -0.9 for PG and 0.14 for PG/16% SiC at room temperature in the a/b mode.

c. Room Temperature Properties Summary

The room temperature mechanical properties data obtained in this investigation on PG and PG/SiC are summarized and compared in Table II. It is apparent that the SiC addition significantly reduces the anisotropy of PG and greatly strengthens it in the c direction. No shear measurements were made. However, it appears certain that the SiC addition increases the shear strength in the ab plane.

d. Flexural Strength Data

Some limited room temperature flexural strength and elastic modulus data were obtained in August 1972 at Atlantic Research on PG/SiC ring segments cut from the ends of materials properties specimens intended to be submitted to SoRI and or plate. The PG/SiC microstructure of those specimens examined showed a fine dispersion of SiC needles in a PG matrix. All specimens were broken with the substrate side in tension. The details

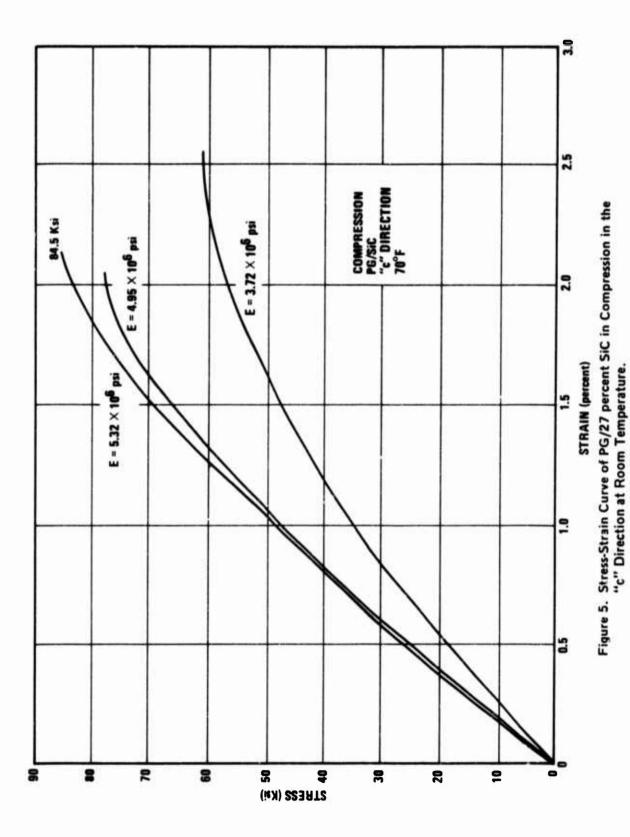


Table II. Room Temperature Mechanical Properties.

		PG	PG 20% SiC	RATIO
ab	ELASTIC MODULUS, TENSION, psi \times 10^6	3.6	4.6*	1.3
C	ELASTIC MODULUS, COMPRESSIVE, psi × 10 ⁶	1.5**	4.7	3
	RATIO ab/c	2.4	1.0	
ab	TENSILE STRENGTH, psi	9000	25,000	3
C	TENSILE STRENGTH, psi	300	8000**	10
	RATIO ab/c	11	3	
	POISSON'S RATIO ab/ab	0.12	0.14*	1
ab	TENSILE STRAIN TO FAILURE, percent	0.25	0.5	2

^{*}BOTH TENSION AND COMPRESSION

^{**}PR!VATE COMMUNICATION FROM SRI

are contained in Appendix III. A small correction was made for the 1 inch radius of curvature. The flexural strengths are plotted in Figure 6 as a function of SiC content. As expected, the data is somewhat higher than the room temperature tensile strength data (see Figure 3). There appears to be a slight trend toward higher strengths at higher SiC content. However, the slope of the dotted line was influenced by the tensile data trend.

The room temperature modulus of elasticity was also calculated using the deflection measurement at failure. The data is plotted in Figure 7 along with similar data from the tensile tests. The similarity of data in the flexural and tensile modes is quite good.

A number of NOL ring tests were also made at room temperature on PG/SiC. However, this approach was abandoned when it was determined that the strengths were only 20 to 40 percent of that obtained in the flexural tests.

SECTION IV

DATA SUMMARY

The	ermal Expansion	Figure 1
The	ermal Conductivity	Figure 2
Ten	sile Strength – a direction	Figure 3
Ten	sile Elastic Modulus — a direction	Figure 4
Ten	asile σ€ Curves - a direction (typical)	Appendix B - Figures 13 and 14
Con	npressive Elastic Modulus - a direction	Appendix B - Figure 16
Cor	mpressive o-e Curve - a direction (typical)	Appendix B - Figure 18
Con	npressive $\sigma \in Curve - c$ direction	Figure 5
	nparison of Room Temperature Properties PG and PG/SiC	Table II

SECTION V

CONCLUSIONS

1. The addition of 20 weight percent SiC to PG renders the material nearly isotropic with respect to thermal expansion (first cycle) and modulus of elasticity.

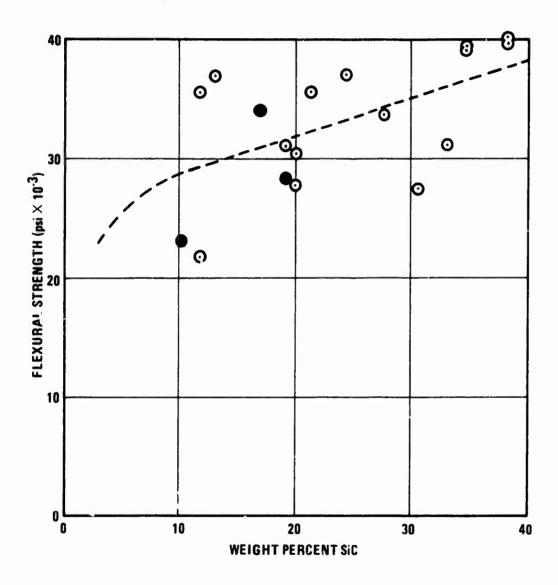


Figure 6. Flexural Strength of PG/SiC in the "ab" Plane at Room Temperature.

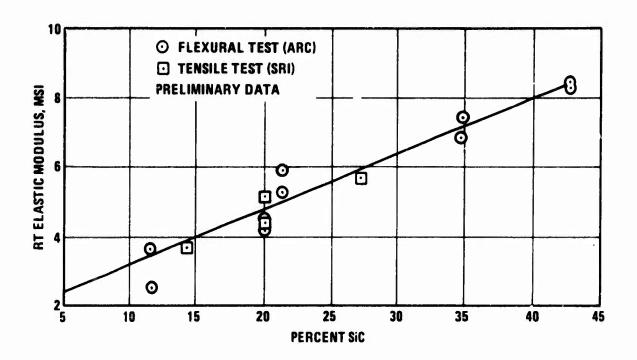


Figure 7. Modulus of Elasticity in Flexure and in Tension of PG/SiC at Room Temperature.

- 2. The addition of 20 weight percent SiC to PG renders it less anistropic with respect to thermal conductivity. However, the material must still be considered anisotropic with respect to this property.
- 3. The addition of SiC to pyrographite increases its strength and modulus of elasticity in the ab plane up to 4000°F.

SECTION VI

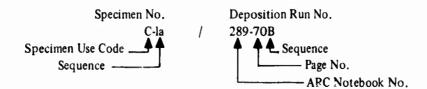
RECOMMENDATION

A much more detailed and comprehensive set of property data should be procured using PG/SiC codeposit material fabricated in large lots so that replicate specimens can be fabricated that are identical with respect to SiC concentration and morphology.

SECTION VII

MATERIAL FABRICATION

A system was established so that all coated parts could be catalogued. Each specimen carries two series of numbers. The first identifies the specimen use, in this case, characterization. The second series denotes the deposition run log book number. An example of this system as used in this report is as follows:



This fabrication description covers only those coating materials which were actually used for testing at SoRI.

1. THERMAL PROPERTY MATERIAL

All thermal property coating materials were fabricated as free standing bodies. To achieve this, the following procedure was used: For the PG/SiC parts, a precoat of unalloyed PG, 5 to 10 mils thick, is deposited on ATJ substrate at 3700°F. Deposition is then stopped for one-half hour and the temperature is reduced to 3200°F. At this time the deposition gas mixture is introduced and the desired coating thickness is applied. After cooling and disassembly, the substrate is machined longitudinally in four places to the coating. The substrate can then be readily separated from the coating, as no bonding takes place between the PG and the PG/SiC.

The unalloyed free-standing PG parts are fabricated in a similar manner; the precoat and the final coat are both applied at 4000°F and the substrate is AGSR graphite. The substrate is slotted longitudinally before deposition leaving a substrate thickness of 30 to 50 mils in four places, 90 degrees apart. This allows the substrate to break apart during cooldown and aids in the prevention of cracking of the PG coating.

The coatings were deposited in both cylindrical and plate form. The cylindrical substrates were 2 inches I.D. by 3.75 inches O.D. by 3.5 inches long. Three of these graphite cylinders were placed in series inside of a 4-inch I.D. tubular resistance heated Pereney furnace as shown in Figure 8. Only the central unit was used for SoRI property specimens except, on occasion, a shorter length was used from the downstream section. The plates were made in a similar manner, as shown in Figure 9, except that the inside surface of the substrate was broached to form a square, instead of circular, cross section. The saw cuts were all made in the corners. The aft plates, 2 1/4 inches wide, were submitted to SoRI for property measurement.

The fabrication parameters are listed in Table III. The reactants were introduced in quite low concentrations in nitrogen carrier gas. For PG/SiC, the total flow rate was 140 scfh, giving a velocity at coating temperature of 150 and 90 ft/sec for the circular and square cross-sections, respectively. For PG, the velocities were 60 percent greater.

The methyl trichlorosilane, MTS, flow rate was controlled by bubbling metered nitrogen through a container of liquid MTS held at 90°F. Using the calculation method described in Phase I, Task 1, and assuming saturation of the bubbler gas with MTS, the MTS concentration was varied, as shown in Table III, from 0.45 to 0.98 percent. The methane CH₄ concentration was maintained at near 2.9 percent for PG/SiC and 2.0 percent for PG. Hydrogen H₂ was used at a near 6 percent level in most of these PG/SiC runs. It was later abandoned. It appeared to be ineffective. The water-cooled copper injector contained one single influent hole, through which all of the reactant gases were introduced, and which was maintained approximately 4 inches from the fore end of the substrate section being coated for properties measurement. The coating densities (by sink-float), the SiC contents (by ashing), and the thicknesses (by microscopic filar measurement of polished end pieces) were all measured at Atlantic Research immediately after coating and prior to shipment to SoRI. In addition, the morphology was determined by microexamination of polished specimens.

A number of densities were measured at Atlantic Research on some of the tested specimens in June 1973 after they were returned. These results are compared with the original 1972 data in Table IV. The close checks are gratifying.

The characteristics of the coating specimen material used at SoRI are summarized in Table V. The last column lists the composition as determined from the density-composition curve developed in Phase I, Task 1. These last column figures are more reliable than the %Sic by ashing data. The low results by ashing are likely caused by incomplete conversion of SiC to SiO₂.

The microstructures of the coating material are summarized in Table VI. Although no microstructure code existed in 1972, some of the specimens were coded in June 1973. The majority of the material showed a fine, evenly dispersed, Code 20, microstructure. A typical Code 20 microstructure, as determined in 1972, is shown in Figure 10. The meaning of the code is given at the bottom of Table VI.

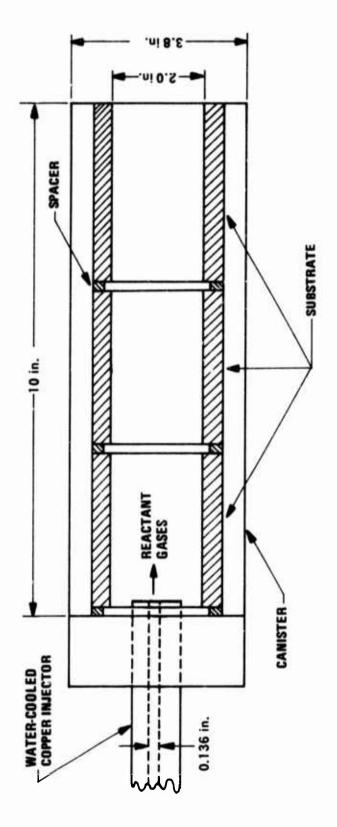


Figure 8. Deposition Assembly for Cylindrical Thermal Property Specimen Fabrication in a 4-inch I.D. Resistance Furnace.

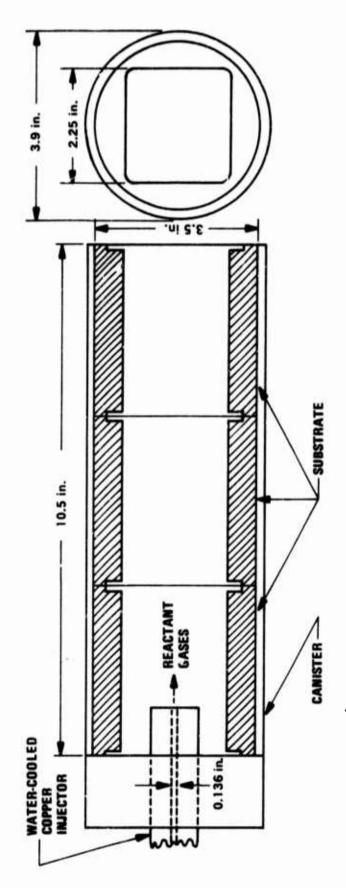


Figure 9. Deposition Assembly for Plate Thermal Property Specimen Fabrication i., a 4-inch i.D. Resistance Furnace.

Table III. Thermal Property Materials Fabrication Parameters and Characteristics.

NO. SHAPE TO SRI (in) CH4 MTS H2 52-18 TUBE 4.75 2.0 0 0 52-30 PLATE 3.5 2.0 0 0 52-30 PLATE 3.5 2.9 0.45 6.0 52-11 TUBE 3.25 2.9 0.45 6.0 52-12 TUBE 3.25 2.8 0.75 5.8 52-12 TUBE 3.25 2.8 0.45 6.0 52-12 TUBE 3.25 2.8 0.45 6.0 52-12 TUBE 3.25 2.8 0.45 6.0 52-2 TUBE 3.5 2.8 0.45 6.0 52-4 TUBE 3.5 3.0	NOMINAL	RAW	LOG	RAW	RAW MATERIAL LENGTH	PE	PERCENT		INJECTOR TO SUBSTRATE	DEN	DENSITY (g/cc)	(B)	X SiC	% Sic By Ashing	IING	THIC	THICKNESS (mil)	Ĩ
15.18 TUBE 4.75 2.0 0 4.5 2.20 -	Sic	PO.	€	SHAPE	TO SRI (in)	CH4	MTS	H ₂	(in)	ENTR.	MIO.	ЕХП	ENTR	1	EXIT	ENTR.	1	EXI
2.18 TURE 4.75 2.0 0 4.5 2.20 -	ERMAL	EXPANSION																
15.30 PLATE 3.5 2.0 4.5 2.20 2.20 2.20 2.20 2.20 2.20 2.20 2.20 2.20 2.20 2.25 2.24 2.25	63	C 2	162-18	TUBE	4.75	2.0	0	0	3.75	2.20	1	2.19	ı	1	ı	29	1	28
C19		C14	162-30	PLATE	3.5	2.0	0	9	4.5	2.20	2.20	2.20	ı	ı	ı	53	1	11
12.1 TUBE 3.5 2.9 0.45 6.0 4.5 2.28 2.3 1.4 116 114 110 112 12.1 TUBE 3.25 2.9 0.45 6.0 3.5 2.4 2.3 2.4 2.9 2.4 2.9 2.4 2.9 2.4 2.9 2.5 2.6 2.5 2.5 2.5 2.5 2.5 3.9 2.7 2.7 2.5 3.0 2.7 2.9 7 2.7 2.2 2.2 3.0 2.2 2.2 3.5 2.5 2.7 2.5 3.0 2.7 2.2 3.0 2.7 2.2 2.7 2.2 2.2 3.0 3.5 2.2 2.7 2.2 2.7	60	C19	289-718		3.5	2.9	0.45	6.0	3.5	2.25	2.25	2.25	8 .3	9.3	8.3	50 60 70	78	76
12.16 11.08E 3.25 2.9 0.45 6.0 3.5 2.35 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 20.3 - 2.4 2.3 2.2 2.4 2.5 2.4 2.5 2.4 2.5 2.4 2.5 2.4 2.7 2.7 1.1 - 1.1 - 1.1 - 1.1 - 1.1 - 1.1 - 1.1 - 1.2 1.2 2.2 2.2 1.1 1.1 1.1 - 1.1 1.1 1.1 1.1 1.1	35	67	289-76	PLATE	3.5	2.9	0.45	9.0	4.5	2.28	2.28	2.3	14.7	18.6	14.4	110	112	5
12-26 PLATE 3.5 2.9 0.60 5.9 4.5 2.4° 2.36 2.25 2.2 <th< td=""><td>20</td><td>C17</td><td>162.11</td><td>TUBE</td><td>3.25</td><td>2.9</td><td>0.45</td><td>6.0</td><td>3.5</td><td>2.35</td><td>1</td><td>2.4</td><td>20.9</td><td>ł</td><td>24.2</td><td>ä</td><td>ı</td><td>126</td></th<>	20	C17	162.11	TUBE	3.25	2.9	0.45	6.0	3.5	2.35	1	2.4	20.9	ł	24.2	ä	ı	126
12-12 TUBE 3.25 2.8 3.5 2.52 - 2.5 3.09 - 29.1 77 - 12-12 ⁴ TUBE 3.25 2.8 0.75 5.8 3.5 2.4 - - 27.4 - - 114 - 13-25 1.9 0.75 5.8 3.5 2.18 2.18 2.18 - - 114 - 13-2 1.0 0 - 2.18 2.18 2.18 - 18.7 - 19.0 - 119 - 110 - - 110 - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 - - 110 -	20	625	162-26	PLATE	3.5	2.9	0.60	5.9	4.5	2.4	2.38	2.35	22.9	22.0	19.8	1	ı	=======================================
13-124 TUBE 3.25 2.8 0.75 5.8 3.5 2.4 - - 27.4 - - 114 - 13-25 TUBE 3.25 2.8 0.45 6.0 - 2.18 2.18 2.18 - - 18.7 110 - 18.0 15.5 12-7 TUBE 3.25 2.9 0.45 6.0 3.5 2.3 ⁺ - 2.35 ⁻ 17.3 - 18.7 110 - 12-2 TUBE 3.25 2.8 0.45 5.9 3.5 2.3 ⁻ 2.35 ⁻ 17.3 - 18.7 110 - 12-4 PLATE 3.5 2.8 4.5 2.48 2.45 2.4 27.8 24.9 2.1 160 152 12-5 TUBE 3.5 2.9 0.45 6.0 3.75 2.42 - 27.6 2.7 14.7 14.7 14.7 14.4 110 112 <td>52</td> <td>C13</td> <td>162.12</td> <td>TUBE</td> <td>3.25</td> <td>2.8</td> <td>0.75</td> <td></td> <td>3.5</td> <td>2.52</td> <td>1</td> <td>2.5</td> <td>30.9</td> <td>1</td> <td>29.1</td> <td>11</td> <td>ı</td> <td>110</td>	52	C13	162.12	TUBE	3.25	2.8	0.75		3.5	2.52	1	2.5	30.9	1	29.1	11	ı	110
13-25 2.1 0 0 - 2.18 2.18 2.18 -	22	C15	162-12	TUBE	3.25	2.8	0.75	80	3.5	2.4	1	ı	27.4	1	ı	11	ı	ı
13-25 PLATE 13 2.1 0 - 2.18 2.18 2.18 2.18 2.18 2.18 - - - 180 125 12-7 1 UBE 3.25 2.9 0.45 6.0 3.5 2.3 ⁺ - 2.35 ⁻ 17.3 - 18.7 110 - 12-2 1 UBE 3.25 2.8 0.45 5.9 3.5 2.3 ⁻ - 2.35 ⁻ 17.3 - 18.7 110 - 12-4 PLATE 3.5 2.8 4.5 2.48 2.45 2.47 27.8 2.49 152 - 141 165 12-5 1.0BE 4.5 3.0 0.77 0 3.75 2.42 - 27.6 - - 141 165 18-7 1.0BE 3.5 2.9 0.45 6.0 4.5 2.28 2.27 - - 27.6 - - 141 165	ERMAL	CONDUCTIVI	ΙŢ															
7.2.7 TUBE 3.25 2.9 0.45 6.0 3.5 2.3 ⁺ - 2.35 20.6 - 18.7 110 - 12.2 TUBE 3.25 2.8 0.45 5.9 3.5 2.3 ⁻ - 2.35 17.3 - 19.8 134 - 12.42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 152 - 152 - 152 - 152 - 152 - 152 - 12.8 152 14.1 165 152 - 141 165 - 141 165 - 141 165 - - 141 165 - - 141 165 - 141 165 - - 141 165 - 141 165 - - 141 160 152 - - - - - <		C38	033-25	PLATE	13	2.1	0	0	ı	2.18	2.18	2.18	ı	ı	I	180	927	2
12-2 TUBE 3.25 2.8 0.45 5.9 3.5 2.3 ⁻ - 2.35 ⁻ 17.3 - 19.8 134 - 12-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 22.1 160 152 12-50 TUBE 4.5 3.0 0.77 0 3.75 2.42 - - 27.6 - - 141 165 19-76 PLATE 3.5 2.9 0.45 6.0 4.5 2.28 2.3 14.7 18.6 14.4 110 112 12-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 22.1 160 152	20	C11	162.7	TUBE	3.25	2.9	0.45	6.0	3.5	2.3+	1	2.35	20.6	1	18.7	110	1	176
12-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.47 27.8 24.9 22.1 160 152 12-50 TUBE 4.5 3.0 0.77 0 3.75 2.42 - - 27.6 - - 14.1 165 19-76 PLATE 3.5 2.9 0.45 6.0 4.5 2.28 2.37 14.7 18.6 14.4 110 112 12-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.45 2.4 27.8 24.9 22.1 160 152	20	020	162.2	TUBE	3.25	2.8	0.45	5.9	3.5	2.3	ł	2.35	17.3	1	19.8	¥	I	150
72-50 TUBE 4.5 3.0 0.77 0 3.75 2.42 - - 27.6 - - 141 165 19-76 PLATE 3.5 2.9 0.45 6.0 4.5 2.28 2.37 14.7 18.6 14.4 110 112 12-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 22.1 160 152	25	623	162-42	PLATE	3.5	2.8	0.98		4.5	2.48	2.45	2.4	27.8	24.9	22.1	160	152	<u>¥</u>
19-76 PLATE 3.5 2.9 0.45 6.0 4.5 2.28 2.28 2.3 14.7 18.6 14.4 110 112 12.24 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 22.1 160 152	52	C47	162-50	TUBE	4.5	3.0	0.77	0	3.75	2.42	1	1	27.6	ı	ı	Ē	165	167
C7 289-76 PLATE 3.5 2.9 0.45 6.0 4.5 2.28 2.28 2.3 14.7 18.6 14.4 110 112 C31 162-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 22.1 160 152	ECTRICA	NL RESISTIVI	Δ															
C31 162-42 PLATE 3.5 2.8 0.98 5.8 4.5 2.48 2.45 2.4 27.8 24.9 22.1 160 152	15	C2	289.76	PLATE	3.5	2.9	0.45	6.0	4.5	2.28	2.28	2.3	14.7	18.6	14.4	110	112	961
	52	G31	162-42	PLATE	3.5	2.8	0.98	5.	4.5	2.48	2.45	2.4	27.8	24.9	1.22	160	152	<u>¥</u>

***USED FOR HALF OF AFT SECTION, INSTEAD OF USUAL MIDDLE SECTION.**

Table IV. Density Rechecks — Thermal Properties.

NOMINAL	RAW MATERIAL	OLE	OLD DENSITIES		NEW RECHECK DENSITIES				
%SiC	NO.	ENTR.	MID.	EXIT	ENTR.	MID.	EXIT	PROP	ERTY
20	C17	2.35		2.4	2.35	_	2.4	CTE	8b
25	C13	2.52	_	2.5	2.48	_	2.48	CTE	ab
25	C15	2.4	_	_	2.38	_	_	CTE	C
20	C11	2.3+	_	2.35	_	2.31ª	_	k	ab
20	C11	2.3+	-	2.35	_	2.31 ^b	-	k	C
25	C29	2.48	2.45	2.40	FIVE PLA 2.48, 2.42			k	ab

aCENTER OF SRI SPECIMEN CR-2AB-C-11

bCENTER OF SRI SPECIMEN CR-1C-C11

CSPECIMEN CONSITED OF FIVE PARALLEL PLATES - USED SPECIMEN

Table V. Thermal Properties - Specimen Characterization Summary.

NOMINAL % SiC	RAW MATERIAL NO.	RAW MATERIAL SHAPE	RAW MATERIAL LENGTH TO SRI (in)	MEASUREMENT DIRECTION	AVERAGE THICKNESS (mil)	AVERAGE % SiC BY ASHING	AVERAGE DENSITY (g/cc)	AVERAGE % SIC FROM DENSITY	TOTAL RUN TIME (hr)	DEPOSITION RATE (mil/hr)
THERMAL	THERMAL EXPANSION					i				
•	23	TUBE	က	ą	92	•	2.20	ı	5.0	55
•	C14	PLATE	2	U	70	•	2.20	1	6.0	12
60	C19	PLATE	3.25	2 & de	70	9.0	2.24	(13)b	9.0	7
15	13	PLATE	3.25	ab & c	110	15.9	2.28	2	11.0	2
2	C17	TUBE	1.5	4	110	23.3	2.35	72	1.7	=
20	C25	PLATE	٣	U	115	21.6	2.35	27	8.0	=
\$2	C13	TUBE	ဗ	đ	95	30.4	2.48	(41)	5.7	11
25	C15	TUBE	1.5	ú	110	27.4	2.38	30	5.7	61
THERMAL	THERMAL CONDUCTIVITY	<u>}</u>								
•	C38	PLATE	9	ą	170	0	ı	ı	14.0	12
20	113	TUBE	m	ab & c	140	19.7	2.31	22	69	91
20	953	TUBE	က	ပ	145	18.5	2.3	12	8.2	5
52	C29	PLATE	8	qe	150	25.3	2.45	(37)	12.0	13
52	C47	TUBE	2	v	160	27.6	2.42	(3	6.0	12
ELECTRIC	ELECTRICAL RESISTIVITY	≥								
15	67	PLATE	4	全	110	15.9	2.28	6	11.0	2
25	C31	PLATE	2	qe	150	24.9	2.42	(34)	12.0	13

aUSE FRONT HALF OF AFT SECTION, INSTEAD OF USUAL MIDDLE SECTION.

^bBracketed values less accurate since outside of composition where density-composition relationship established.

Table VI. Microstructure of Thermal Property Specimens.

RAW MATERIAL NO.	NOMINAL % SiC	SIC NEEDLES	DISPERSION
THERMAL EX	PANSION		
C2	0	FAIRLY GOOD. SOME LARGE CON	ES WITH STRAIN LINES
C14	0	NO RECORD	
C19	8	FINE. CODE 21ª PER JUNE, 1973	GRAIN BOUNDARY CONCENTRATIONS
C7	15	FINE	EVEN
C17	20	FINE. CODE 20	EVEN
C25	20	NO RECORD. CODE 30 ⁸ (somewhat coarse) PER JUNE, 1973	EVEN (per June, 1973)
C13	25	FINE. CODE 20	EVEN
C15	25	FINE. CODE 20	EVEN EXCEPT GRAIN BOUNDARY CONCENTRATIONS NEAR EXIT END
THERMAL CO	NDUCTIVITY		
C38	0	NO RECORD	
C11	20	FINE. CODE 20 PER JUNE, 1973	EVEN
C56	20	FINE	EVEN
C29	25	NO RECORD. CODE 30 ⁸ (somewhat coarse) PER JUNE, 1973	EVEN (per June, 1973)
C47	25	NO RECORD. CODE 30/60 ^a ENTRANCE END AND CODE 11/21 ^a EXIT END PER JUNE, 1973	EVEN ENTRANCE END GRAIN BOUNDARY CONCENTRATIONS AT EXIT END

 $^{^{}a}$ CODE 10 - SiC NEEDLES < 0.9 μ dia. AND L/D > 3. GOOD DISPERSION

CODE 11 - SIC NEEDLES < 0.9 μ dia. ANS L/D >3. POOR DISPERSION - COLLECTS IN GRAIN BOUNDARIES

CODE 20 - SiC NEEDL 58 0.9 TO 2.6μ dia. AND L/D > 3. EVEN DISPERSION

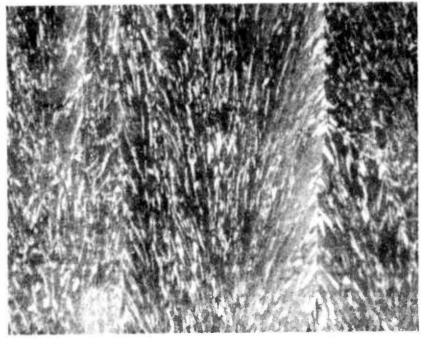
CODE 21 - SiC NEEDLES 0.9 TO 2.6 μ dia. AND L/D > 3. POOR DISPERSION COLLECTS IN GRAIN BOUNDARIES

CODE 30 - SiC NEEDLES > 2.6 μ dia. AND L/D > 3. EVEN DISPERSION

CODE 60 - SIC NEEDLES > 2.6 μ dia. AND L/D > 3. POOR DISPERSION PG STRAIN LINES



Old 1972 (SiC is dark)



160X New 1973 (SiC is light)

Figure 10. Microsturcture of PG/20 percent SiC Thermal Conductivity Specimen from C11 Material Code 20.

2. MECHANICAL PROPERTY MATERIAL

The tensile and compressive specimens were fabricated by coating the inside diameters of 2 inch I.D. graphite cylinders using techniques similar to those described under the thermal property material. Schematics of the coating apparati are shown in Figures 11 and 12. Only the aft compressive test specimen was sent to SoRI. The coating fabrication parameters are shown in Table VII. The pure pyrographite was deposited using 2 percent methane in nitrogen carrier gas at 4000°F. The PG/SiC was deposited using approximately 3 percent methane and 0.2 percent MTS, for the nominally 8 percent SiC composition, and 0.5 percent MTS for the nominally 20 percent SiC composition at 3200°F. The coating thickness profiles are shown in Table VIII. It should be noted that the coating thickness does not become appreciable until a point approximately 5 inches downstream from the injector. The original intention at SoRI was to fabricate the specimens by thinning down the central portions so as to expose the coating but leave the substrate on the ends for mechanical support during testing. However, because of difficulties already discussed in this report, SoRI finally turned to the fabrication of arc coupon specimens per Figure 3 of Appendix II for the tensile specimens (5 inches long) and per Figure 5 for the compression specimens (2.4 inches long). The centers of the specimens were near the center of the tubes supplied to SoRI. No characterization work was done at Atlantic Research prior to shipment to SoRI.

The silicon carbide contents of the specimens were determined at SoRI by ashing using material circumferentially adjacent to the central parts of the specimens. After return of the material, densities and the microstructures adjacent to the fracture were determined at Atlantic Research. The results are shown in Table IX.

The percent silicon carbide was determined at Atlantic Research indirectly from the density, using a density to SiC content relationship developed at Atlantic Research and as described in Phase II, Task 1. As can be seen from the table, these indirect values check the %/SiC by ashing at SoRI quite well. The greatest anomaly appears to be with specimen C45 which analyzed 16 percent at SoRI and 22 percent at Atlantic Research by density. It may be observed in Figure 16 of Appendix II that Specimen C45 had a higher room temperature modulus of elasticity than expected for its composition. The density values shown in Table IX, as determined by SoRI, appear to be inaccurate. The morphology of the material appears to be, on the average, Codes 20 and 21 which is fine silicon carbide needles, 1 to 2 microns in diameter either well dispersed or somewhat concentrated in the grain boundaries.

The pure PG plate used for ab direction tensile specimens was fabricated in the familiar square cross-section using plates 2 1/4 inches wide on their inside surfaces by 4 1/2 inches long. This coating was done in the 6-inch Pereny furnace which is also resistance heated. Certain of the coating parameters and the material characteristics of this material are shown in Table VII and IX, respectively.

The raw material for the compressive testing of PG/SiC in the c direction, which testing was not reported in Appendix II although the testing was done at SoRI, was especially thick plate fabricated in the induction heated 12-inch-diameter furnace under the Scale-up Program. Fabrication parameters, and the material characteristics, are listed in Tables VII and IX, respectively.

The microstructure of PG/SiC material C33 is shown at various magnifications in Figure 13. The microstructure of pure PG material C38 is shown in Figure 14.

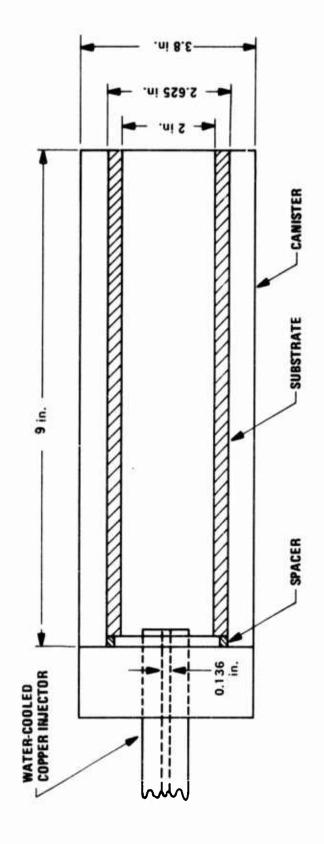


Figure 11. Deposition Assembly for Cylindrical Tesnile Test Specimen Fabrication in a 4-inch I.D. Resistance Furnace.

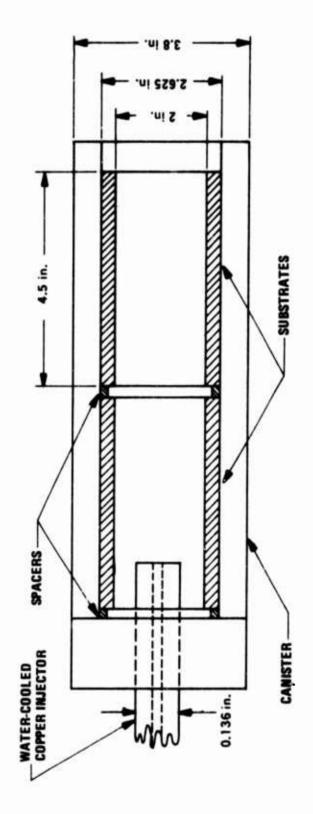


Figure 12. Deposition Assembly for Cylindrical Compressive Test Specimen Fabrication in a 4-inch I.D. Resistance Furnace.

Table VII. Mechanical Property Materials Fabrication Parameters (Cylinders).

NOMINAL % SiC	RAW MATERIAL NO.	LOG RUN NO.	PE CH ₄	RCENT MTS	H ₂	INJECTOR TO SUBSTRATE (in)	TOTAL RUN TIME (hr)	MAXIMUM THICKNESS (near center) (mil)	MAXIMUM COATING RATE (mil/hr)
TENSILE ab									
08	C38	033-25	2.0	_	0	NOT RECORDED	14.0	140	10
8	C53	162-69	2.9	0.19	0	0.5	8.5	168	20
8	C55	162-70	2.9	0.19	0	NOT RECORDED	8.2	162	20
20	C35	162-37	2.9	0.47	6.0	0.5	8.0	168	21
20	C43	162-64	2.9	0.47	6.0	0	9.1	146	16
20	C39	162-59	2.9	0.47	6.0	0	5.0	128	26
COMPRESSI	VE ab								
0	C16	162.46	2.1	_	0	3.75	4.2	59	14
	C18	162-47	2.1	•••	0	3.75	4.2	56	13
8	C51	162-68	2.9	0.19	0	0.5	8.0	164	20
	C59	162-72	2.9	0.19	0	3.75	8.2	166	20
20	C33	162-38	2.9	0.47	6.0	4.0	8.0	179	22
	C45	162-65	2.9	0.46	6.0	4.5	6.25	110	18
	C23	162-16	2.9	0.61	6.0	3.75	7.0	177	25
	C21	162-15	2.9	0.46	6.0	3.75	7.8	176	23
	C49	162-67	2.9	0.46	6.0	4.5	6.4	113	18
TENSILE c									
0	C30	162-76	2.1	-	0	0	3.2	56	17
8	C53		SEE A	ABOVE	UNDE	R TENSILE ab			
20	C39		SEE A	BOVE	UNDE	R TENSILE ab	•		
COMPRESSI	VE c								
20		001-47	2.8	0.34	0	-	17.8	261	15

aPG PLATE

Table VIII. Thickness Profiles (mil) Mechanical Property Specimen.

MATERIAL NO.	ENTRANCE	1"	<u>2"</u>	3"	4"	5"	6"	7"	<u>8"</u>	EXIT
TENSILE ab	7									
C38ª	160/200	-	110/145	_	90/110	_	-	_	_	_
C53	53	62	93	148	168	167	144	120	_	90
C55	63	89	125	155	162	152	123	_	-	85
C35	56	71	102	141	168	173	152	132	105	87
C43	18	30	47	70	106	134	146	133	114	88
C39	5	15	29	51	95	116	128	125	108	86

COMPRESSIVE ab

RAW MATERIAL NO.	ENTRANCE	1"	2"	3"	4"	EXIT	5"	6"	EXIT (C51 only)
C16	44	50	57	59	_	49	_	_	-
C18	34	46	53	56	_	54	-	_	-
C51 ^b	67	86	138	155	164	_	140	118	81
C59	161	169	166	141	104	82	_	-	-
C33	87	149	178	179	154	140	-	-	-
C45	106	115	110	95	-	72	_	_	-
C23	167	177	173	150	121	90	_	_	-
C21	170	182	176	159	129	107		-	-
C49	107	116	113	94	-	71	-	-	-

^aPLATE 2 1/4 in. X 4 in. LONG

bORIGNIALLY FABRICATED AS A TENSILE SPECIMEN

Table IX. Mechanical Properties - Specimen Characterization Summary.

NOMINAL	RAW MATERIAL NO.	% SIC BY ASHING	DENSITY (g/cc)		% SiC FROM	
% SiC			SRI	ARC	ARC DENSITY	MORPHOLOGY*
TENSILE ab						
0	C38	0	2.19	2.18	_	_
8	C53	13	2.37	2.21	12	CODE 21
8	C55	16	2.37	2.21	12	CODE 10 EXCEPT L/D <3
2 G	C35	20	2.44	2.31	22	GROUP 20
20	C43	20	2.41	-	-	-
20	C39	27	2.48	-	-	-
COMPRESSIV	E ab					
0	C16	0	_	-	-	_
	C18	0	-	-	-	-
8	C51	16	***	2.24	15	CODE 11
	C59	16	2.34	2.24	13	CODE 10/20
20	C33	13	2.34	2.21	10	CODE 20
	C45	16	_	2.31	22	CODE 21
	C23	20	_	2.30	21	CODE 30
	C21	25	2.43	2.34	26	CODE 20
	C49	25	-	2.325	24	CODE 21
TENSILE c						
0	C30	0		-	_	-
8	C53	13	***	wards	-	-
20	C39	27	-	~	-	-
COMPRESSIV	E c					
20	-	26	_	2.35	27	CODE 30/21

^{*}SEE TABLE V FOR CODE.

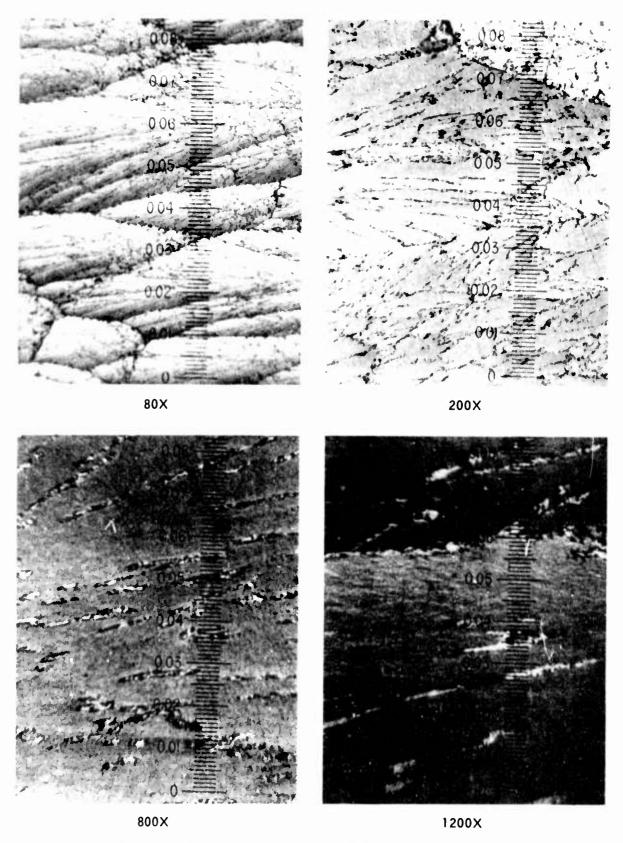
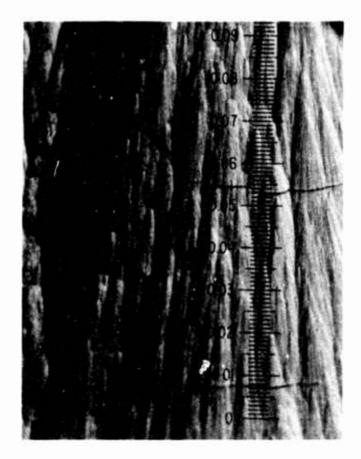


Figure 13. Microstructure of PG/13 percent SiC Compressive Specimen from C33 Material Code 20.



80X

Figure 14. Microstructure of Pure Pylog/aphite Tensile Specimen from C38 Material.

SECTION VIII

DISCUSSION

Thermal expansion measurements are generally made over fairly long periods of time. The specimens are heated slowly between temperature increments, and the strain is measured optically or with the use of a dilatometer. However, in the nozzle application, short-time thermal expansion data is required. It is now well known that PG/SiC is unstable above 4000°C. Note the permanent deformation in Figures 3 through 12 in Appendix I. The permanent deformation, or instability, is probably caused by an ordering of the pyrographite phase and possibly by a conversion of the cubic beta silicon carbide into hexagonal alpha silicon carbide. The ordering of the PG causes a growth in the ab plane and a shrinkage in the c direction.

The permanent deformations in the thermal expansion measurements made at SoRI are summarized in Table X. A more realistic picture of the true short-time expansion above 4000° F might be obtained by subtracting these deformations from the first cycle $\Delta L/L$ data, as shown in the corrected column at 5000° F. After application of the permanent deformation correction, the $\Delta L/L$ versus temperature anisotropy is considerably increased, as seen in Table XI. This is as expected in view of the pyrographite ordering phenomenon.

It is important to note that this thermal ordering in the pyrographite phase phenomenon masks the true shape of the thermal expansion curve. A second cycle slow thermal expansion curve might, in fact, more closely simulate a first cycle rapid heatup thermal expansion cu.ve. A second cycle curve was obtained for pure pyrographite at SoRI, as shown in Figure 3 of Appendix I. This could have been predicted in at least an approximate way by subtracting the permanent deformation, which was approximately 1.5 percent from the first cycle data, from the first cycle curve.

It may then be somewhat realistic with respect to a thermal stress calculation during a nozzle firing to consider that the thermal expansion anisotropy of pyrographite silicon carbide is really very close to that shown in Table XI after the permanent deformation correction has been applied. The material then may be considerably more anisotropic than originally thought. It is likely, however, that the first cycle data applies quite well when cooldown stresses in the coating furnace are being considered. The psuedo second-cycle data should possibly be considered only for test firing structural analyses.

Extrapolation of the elastic modulus versus the percent silicon carbide in Figure 7 to 0 percent SiC indicates an elastic modulus for the pyrographite phase of approximately 2.5 million psi. The Phase I, Task 1 results indicated a density for the PG phase of 1.14 gm/cc. It would seem very desirable to be able to improve the density and quality of the pyrographite phase. It would appear unlikely that the SiC phase could be significantly improved unless its activity could be tied up chemically, and its dissociation pressure reduced thereby, by addition of another metal carbide so as to form a compound or a solid solution.

Some loss of SiC occurred in the 4500°F mechanical property tests. The strength and elastic modulus data at 4500°F are conservative, i.e., if anything the values should be higher. It is doubtful that PG or PG/SiC would exhibit a strong strain rate effect, although it has been observed that the strength of PG is decreased somewhat by increasing strain rate, e.g., a 20 percent reduction caused by a 100-fold increase in strain rate.

Table X. Permanent Deformation in Thermal Expansion Testing of PG/SiC.

PERCENT SiC	PERMANENT DEFORMATION PERCENT 5000°F PD	PERCENT △L/L 4000°F	PERCENT ^L/L 5000°F ^	PERCENT CORRECTED △L/L 5000°F △-PD
ab DIRECTION				
0	1.5	0.75	2.25	0.75
8	0.32	0.98	1.34	1.02
18	0.36	1.05	1.42	1.06
27	0.35	1.11	1.51	1.16
40	0.20	1.16	1.54	1.34
c DIRECTION				
0	-2.8	5.0	3.5	6.3
8	-1.0	1.5	1.5	2.5
18	-0.35	1.32	1.62	1.94
27	-0.30	1.24	1.50	1.80
30	-0.42	1.25	1.45	1.87

Table XI. Thermal Expansion Anisotropy of PG/SiC.

PERCENT SiC	FIRST CYCLE ANISOTROPY 5000°F	FIRST CYCLE ANISOTROPY 5000°F CORRECTED FOR PERMANENT DEFORMATION®
0	1.55	8.4
8	1.12	2.5
18	1.14	1.8
27	0.99	1.55

acorrected anisotropy = $\frac{\triangle L/L \cdot PERMANENT DEFORMATION ("c" direction)}{\triangle L/L \cdot PERMANENT DEFORMATION ("ab" direction)}$

All of the data given in this report should be redetermined. A technique should be developed such that a large quantity of PG/SiC of uniform concentration, thickness and morphology could be produced. This would represent a tremendous saving in coating run time and coating evaluation time and would yield a more reliable set of properties data. Specifically, large plates or large cylinders should be produced. A minimum leady of 5 inches, with a uniform 3-inch center section, is desirable because of the tensile specimen requirement.

APPENDIX I

THERMAL PROPERTIES SORI

THERMAL PROPERTIES OF PYROLYTIC GRAPHITE AND CODEPOSITED SIC/PG WITH VARIOUS WEIGHT PERCENTAGES OF SILICON-CARBIDE

Final Report

to

ATLANTIC RESEARCH CORPORATION Alexandria, Virginia

Purchase Order 78119

Southern Research Institute Birmingham, Alabama January, 1973

> SORI-EAS-73-031 Project 2931-F

THERMAL PROPERTIES OF PYROLYTIC GRAPHITE AND CODEPOSITED SIC/PG WITH VARIOUS WEIGHT PERCENTAGES OF SILICON-CARBIDE

INTRODUCTION

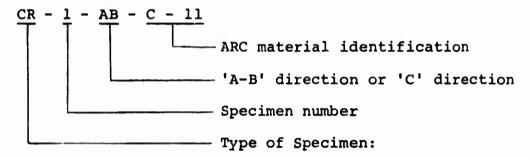
This is the final report to Atlantic Research Corporation covering thermal property evaluations on Pyrolytic Graphite and Codeposited SiC/PG with various weight percentages of silicon-carbide. The evaluations were performed under Purchase Order 78119.

MATERIAL

The material was furnished by Atlantic Research Corporation. We received Pyrolytic Graphite and Codeposited SiC/PG with 8%, 15%, 20% and 25% by weight percentage silicon-carbide.

The test matrix is shown in Table 1.

The specimen were identified at SoRI as follows:



CR - Comparative Rod

RI - Radial Inflow

ER - Electrical Resistivity

TE - Thermal Expansion

X-ray diffraction analysis gave the following values for ARC-PG:

$$d_{002} = 3.438 \times 10^{-8} \text{ cm}$$

$$l_{C} \text{ (crystallite height in the 'C' Direction)}$$

$$= 94 \times 10^{-8} \text{ cm}$$

$$0 = 2.205 \text{ gm/cm}^{3}$$

APPARATUSES AND PROCEDURES

Several apparatuses were used in this program to evaluate thermal expansion, thermal conductivity and electrical resistivity on Pyrolytic Graphite and Codeposited SiC/PG with various weight percentages of silicon-carbide. Each apparatus is briefly described under proper headings, and detailed descriptions of these apparatuses are included in the appendices.

THERMAL EXPANSION

Quartz Dilatometer

Thermal expansion was measured from 70F to 1700F in a quartz tube dilatometer. The dilatometer consists of a hollow quartz cylinder which contains the specimen and a quartz push rod. The bottom of the push rod rested on the top of the specimen and the top of the push rod was flush with the top of the outer cylinder. A dial gage with its styles resting on the top of the push rod was used to measure the relative motion between the specimen and the dilatometer tube. Thermal expansion measurements in the 'A-B' direction were made using a 3-inch long specimen. However, for the 'C' direction (due to small material thickness) several pieces were stacked together to form a specimen. We have used this stacked specimen configuration for the measurements under other several programs conducted here. This method does not add any additional uncertainty in the measurements than the usual ±3 percent.

Graphite Dilatometer

Thermal expansion measurements are made from 70F to 5500F utilizing graphite dilatometer with a dial gage. The basic principle of operation is the same as the quartz dilatometer. The specimen is radiantly heated in a graphite tube furnace.

The uncertainties in measurements of the quartz and graphite dilatometers, based on error analyses, are estimated at ±3 percent and ±5 percent, respectively.

THERMAL CONDUCTIVITY

Comparative Rod Apparatus

The thermal conductivity measurements in the 'A-B' direction were made using a cylindrical specimen. However, to determine thermal conductivity in the 'C' direction, a "bare wire" technique described in the following paragraph was employed.

The method of measuring specimen temperature is illustrated in Figure 1. Chromel/alumel thermocouple wires, 0.005 inch in diameter, were placed through a grooved disc of boron nitride. A double bore alumina tube was placed in this groove to carry the Chromel/alumel wires to the center of the specimen. These wires were flattened at the end to a thickness of about 0.002 inch and positioned on a Grafoil disc which was in contact with the specimen surface. The Grafoil disc served the electrical path to form the thermocouple. The specimen temperature gradient was corrected by substracting the temperature gradient across the Grafoil disc which was obtained from the in-house thermal conductivity data.

Radial Inflow Apparatus

To measure thermal conductivity of SiC/PG sleeve in the 'C' direction, an optical method and the water calorimeter was The configuration for this method is shown in Figure 2. The technique involves measuring inside and outside specimen surface temperatures with an optical pyrometer. The sleeve was centered on a calorimeter by means of ATJ graphite specimens support cylinder fitted into the top and bottom CS graphite quards. In order to view the inner walls of the specimen, no packing was used in the annulus. The calorimeter was painted with a flat black enamel. Heat transfer takes place from the specimen to the calorimeter by gas conduction and radiation. A 0.100 inch diameter hole was drilled in a surrounding cylinder to measure the face temperature of the specimen. This hole provided a blackbody cavity with a length to diameter ratio of about 3:1. A similar hole 0,100 inch in diameter was drilled through both the specimen support cylinder and the specimen. This hole was offset 0.130 inch from the centerline so that the view of the inner wall was not obstructed by the calorimeter.

Electrical Resistivity

Electrical resistivity from 70°F to 1800°F was measured using our electrical resistivity apparatus. The equipment consists of a split furnace, d.c. power supply, d.c. ammeter, k-3 potentiometer and specimen electrodes. The heated zone of the furnace is about 12 inches long which easily allows maintaince of a uniform specimen temperature over 1-1/2 inch specimen gage length.

The uncertainty in measurements of the electrical resistivity with this apparatus is about ±2 percent. This is based on an error analysis of the system as well as con arison runs on materials which gave agreement within about ±2 percent to the values reported in the literature.

DATA AND RESULTS

Thermal Expansion

Thermal expansion data to 5000F in the '\(^1\)-B' and in the 'C' directions of PG and codeposited SiC/PG at four different silicon carbide contents (8%, 15%, 20% and 25% by weight) are presented in Figures 3 through 12 and in Tables 2 through 17. Composite plots for two directions are shown in Figures 13 and 14. Included for a comparison are the across grain expansion data on AGSR graphite which ARC used for the substrate, for the pyrolytic graphite and the with grain data on ATJ graphite (not the same material used by ARC) which was used for the substrate, for the codeposited SiC/PG.

It can be observed from Figures 13 and 14, that with increase in the silicon carbide contents, the thermal expansion in the 'A-B' direction increased while it decreased in the 'C' direction, compared to pyrolytic graphite. Thus, silicon carbide contents reduced the anisotropy. The 20 percent level silicon carbide material was essentially isotropic.

The data indicated that the materials were unstable above about 4200F (approximate temperature of the deposition). At the specimen temperature of about 4200F, the silicon carbide (β -form) dissociates from the pyrolytic graphite. After completion of the runs in graphite dilatometers, we found crystals of silicon carbide deposited on the graphite push rods. (Similar deposition was noticed during the thermal conductivity measurements). This deposition was varified by x-ray diffraction analysis as β -silicon carbide.

One of the specimens from pyrolytic graphite was exposed twice to 5000F. The material shows stability up to 5000F during the second run (see Figure 3).

The ARC-PG did not show negative expansion in the 'A-B' direction from 70F to about 1000F. The negative expansion (rather negative coefficient of expansion) on conventional pyrolytic graphite is explained in terms of Poission contraction of the layer planes (basal) associated with large expansion perpendicular to the layer planes (C-axis) as explained by Riley. Above about 1000F, this effect is assumed to be counteracted by a true therma expansion of the layer planes to produce a small positive thermal expansion coefficient.

¹Riley, D. P., Phys. Soc. 57. 487 (1945).

Why this negative expansion behaviour was not shown by ARC-PG was probably due to the material structure.

It was thought initially that ARC-PG may contain SiC as an impurity. However, x-ray diffraction data indicated that if the impurity of SiC were present, it was less than 1 percent by weight.

The x-ray diffraction study gave 'd' spacing (3.438 Å) which indicated that ARC-PG was turbostratic in structure. (By turbostratic structure we mean that the adjacent basal planes are randomly rotated with respect to one another, and thus, do not display an evidence of three-dimensional ordering. This does not mean that the distance between the layer planes is variable. All the layer planes are at a distance given by 'd' spacing of the x-ray diffraction analysis).

Bacon² and Franklin³ have shown that the increase in the unit cell height 'C' (=2d) above the perfect graphite value of 6.708 A is a quantitative measure of the amount of turbostratic structure. Their relationship is approximated by a parabolic expression:

C = 6.880 - 0.172 (1-p) - 0.128 p (1-p)

where

p = the probability of stacking disorder (basal planes)

Using the value of the 'd' spacing given by the x-ray diffraction, the value of 'p' for ARC-PG was 99 percent. This indicated that the ARC-PG was almost turbostratic in structure. This conclusion is also supported by the value of the crystallite height (ℓ_C). Typical values of 'p' for ATJ-S graphite is 30 percent.

In PG, structure can range from a material where the growth cone develops from the deposition surface (substrate necleated) to a material where the growth cones are regenerated continuously throughout the thickness of the deposition (continuously nucleated). Substrate nucleated structure exhibits maximum crystal orientation and therefore highest anisotropy. Thus, turbostratic structure of ARC-PG indicated a possibility of continuously nucleated structure. Microstructure analysis will varify this observation.

²Bacon, E. E., Acta Crystallite, 4, 558 (1951).

³Franklin, R. E., Acta. Crystallite, 4, 253 (1951).

The high expansion of pyrolytic graphite in the 'C' direction is as expected. As the planes (basal) becomes further apart with temperature, the frequency of vibration is lower (out of plane acoustic modes) due to a decrease in the interplanar interaction. This decrease in the interplanar interaction is due to an increase in the 'd' spacing with temperature.

On an atomic basis, thermal expansion corresponds to an increase in the average interatomic distance. Therefore, comparing microscopic and macroscopic properties one can write:

$$\left(\frac{\frac{d-d}{1}}{d}\right)_{T} = \left(\frac{1-1}{1}\right)_{T} = \left(\frac{\Delta t}{\ell}\right)_{T}$$

where

d = equilibrium distance between two basal planes
 (3.438 Å)

d = above distance at temperature, T

 $\frac{\Delta \ell}{\ell}$ = unit thermal expansion at temperature, T

At 4000F, $\frac{\Delta \ell}{\ell}$ was = 5 x 10⁻³ in./in. or equal to 5 percent

in the 'C' direction. Thus at 4000F, the increased in the basal plane distance was about 5 percent. This increase in the distance between the layer planes resulted in a decrease in the interplanar interaction which caused decreased in the frequency of vibration or an increase in the thermal expansion in the 'C' direction.

More attention was given in explaining the thermal expansion behaviour of ARC-PG, since future improvements in the properties of codeposited material will largely depend upon the properties of this pyrolytic graphite.

Thermal Conductivity

Thermal conductivity data on ARC-PG and Codeposited SiC/PG with 20% and 25% silicon carbide, in the 'A-B' and in the 'C' directions are presented in Figures 15 through 20 and in Tables 18 through 25.

For ARC-PG the anisotropy ratio of conductivity at 200F was 150. The data on ARC-PG for both orientations compares favorably with other SORI data.

At elevated temperatures the thermal conductivity of the undelaminated pyrolytic graphite in the 'C' direction is affected by a prior heat treatment (~3000C or above) but that in the 'A-B' direction, remains at about the same level. to a heat treatment, turbostratic structure is modified so as to exhibit a more 3-dimensional stacking order of the basal planes. Since ordering the crystal structure corresponds to ordering the basal planes, this ordering will increase the interaction energy between the basal planes and therefore will increase the phonon energy. The increase in the phonon energy will result in an increase in the group velocity and therefore, a decrease in the phonon density (population per unit volume), which will lead to a decrease in the thermal resistivity or an increase in the thermal conductivity. However, the structure of each basal plane is not much influenced by a heat treatment. Therefore, at high temperatures, the thermal conduction in pyrolytic graphite in the 'C' direction is generally affected by a heat treatment while that in the 'A-B' direction remain much the same.

Other important factors which will influence the thermal conductivity of pyrolytic graphite such as (1) delaminations and their locations, (2) conduction of gases in the delaminations, and (3) radiation, are discussed elsewhere.

Higher silicon carbide contents have reduced the anisotropy, compared to pyrolytic graphite. The anisotropy ratio of conductivity for 20 percent silicon carbide at 200F was about 10 compared to about 150 for pyrolytic graphite at 200F. A composite plot for two directions are shown in Figures 21 and 22.

Electrical Resistivity

The electrical resistivity data in the 'A-B' direction to 1800F for the codeposited SiC/PG with 15% and 20% silicon carbide are presented in Figure 23 and in Table 26. The data are compared with other pyrolytic graphite. The decrease in the electrical resistivity with temperature is explained in terms of a decrease in the scattering at the crystallite boundary with temperature. In this temperature region (70 to 1800F), the thermal scattering is not that predominant. By increasing the silicon carbide from 15% to 25%, an increase in the resistivity near room temperature by four-fold (4 times) was observed. The electrical resistivity

[&]quot;Thermal conductivity of pyrolytic graphite cylinders in various gas environments." The Ninth Thermal Conductivity Conference, 1969.

of a single crystal silicon carbide near room temperature is about 1,000,000 μ ohm-cm, which explains why an increase in the resistivity occurred with a slight increase in the silicon carbide contents.

Submitted by:

Strepal

S. G. Bapat Associate Engineer

Approved by:

C. D. Pears, Head

Mechanical Engineering Division

SoRI-EAS-73-031 Project 2931-F

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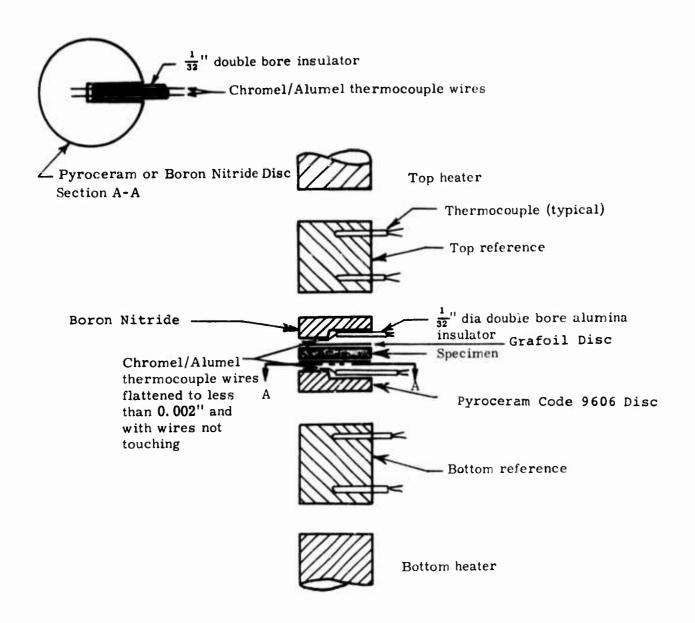
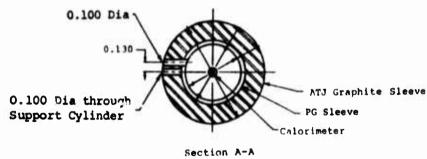


Figure 1. Expanded Schematic of References and Specimen Configuration for Thin Materials Using the Comparative Rod Apparatus and the Bare Wire Method of Measuring Specimen Surface Temperature



Section A-A
Datail of Sight Hole

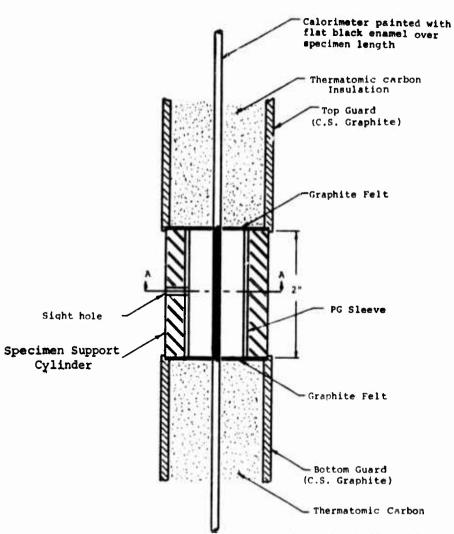


Figure 2. Buildup for Optical Measurements of Inside Surface Temperature of a PG Sleeve during thermal conductivity measurements in the 'C' Direction using the Radial Inflow Apparatus

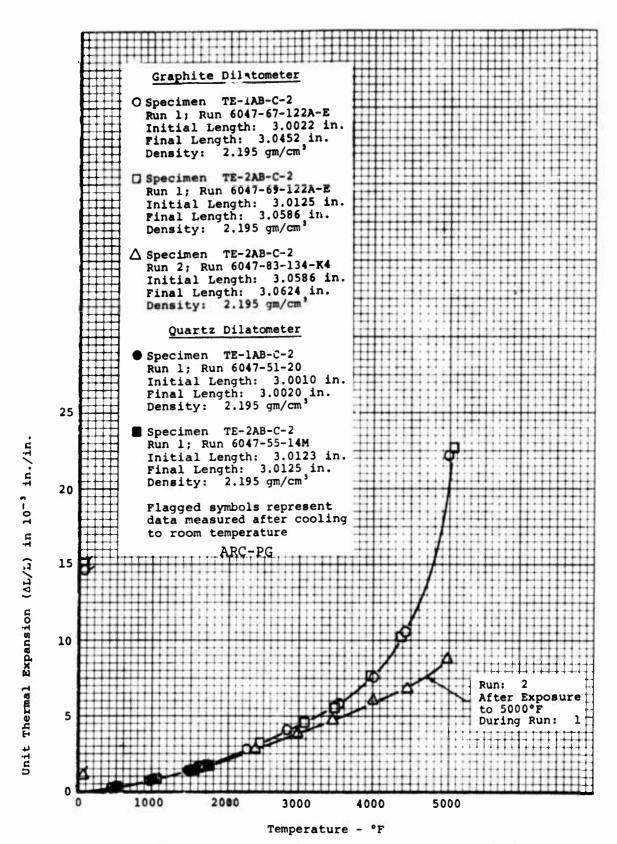


Figure 3. Thermal Expansion of Pyrolytic Graphite in the 'A-B' Direction

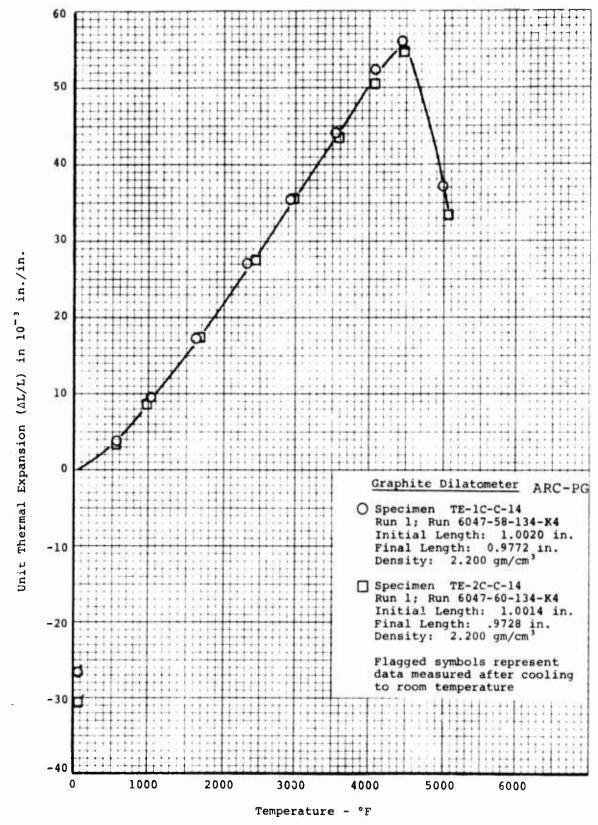


Figure 4. Thermal Expansion of Pyrolytic Graphite in the 'C' Direction

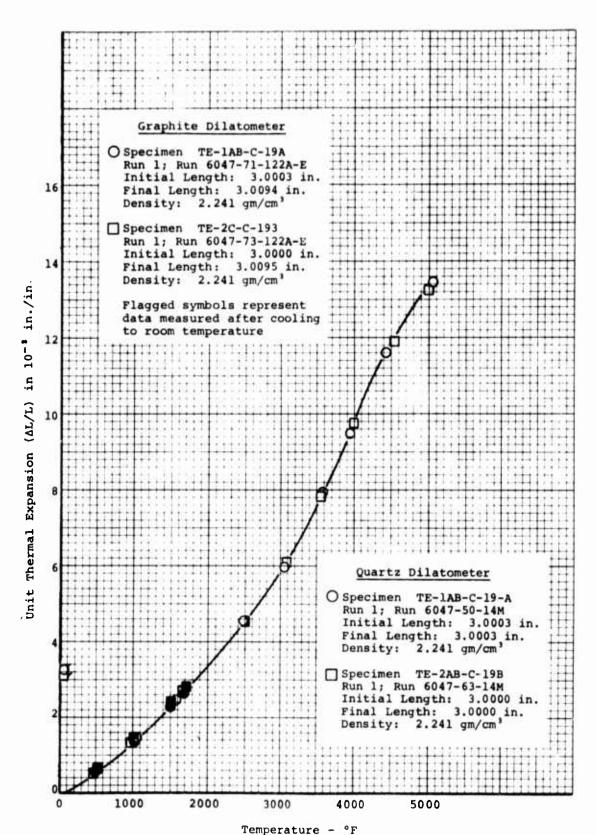


Figure 5. Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 13 Percent SiC in the 'A-B' Direction

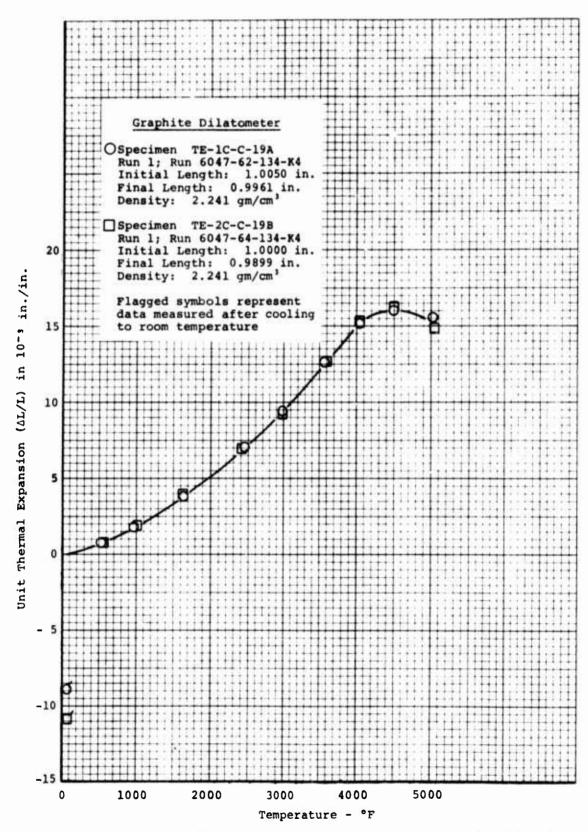


Figure 6. Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 13 Percent SiC in the 'C' Direction

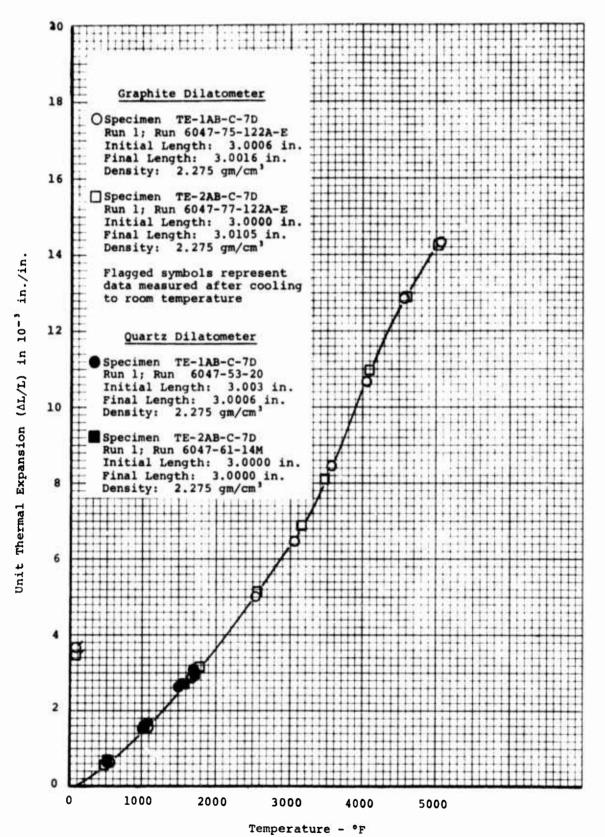


Figure 7. Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 18 Percent SiC in the 'A-B' Direction

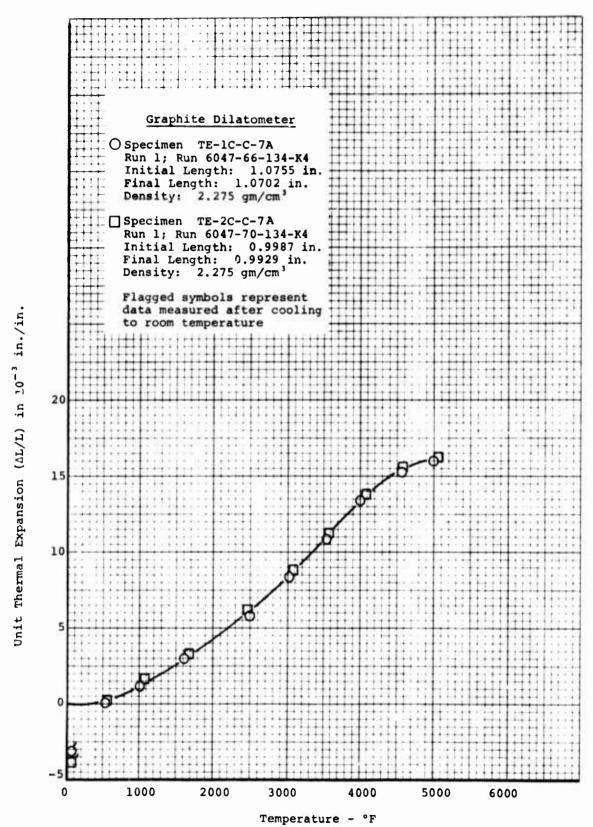


Figure 8. Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 18 Percent SiC in the 'C' Direction

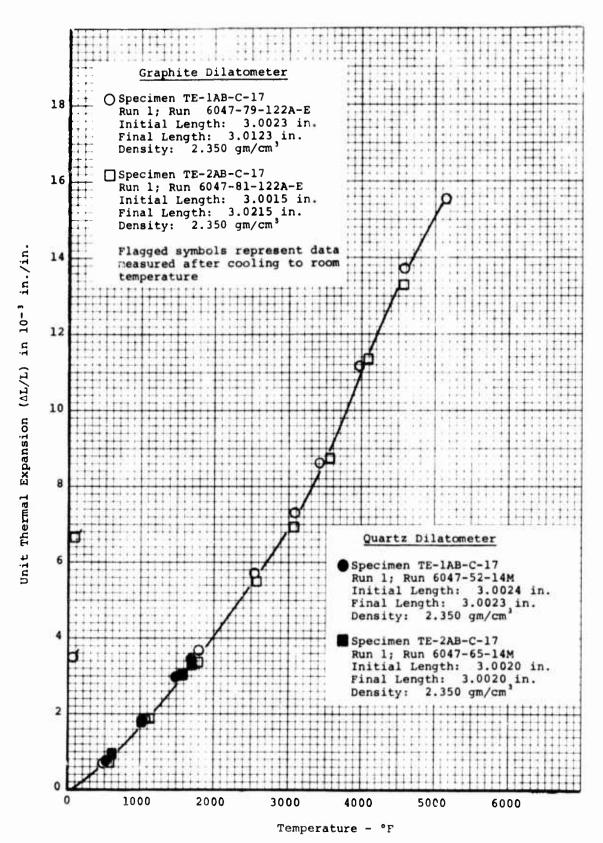


Figure 9. Thermal Expansion of Codeposited Pyrolytic Graphite Sleeve (Coupon Specimen) with 27 Percent SiC in the 'A-B' Direction

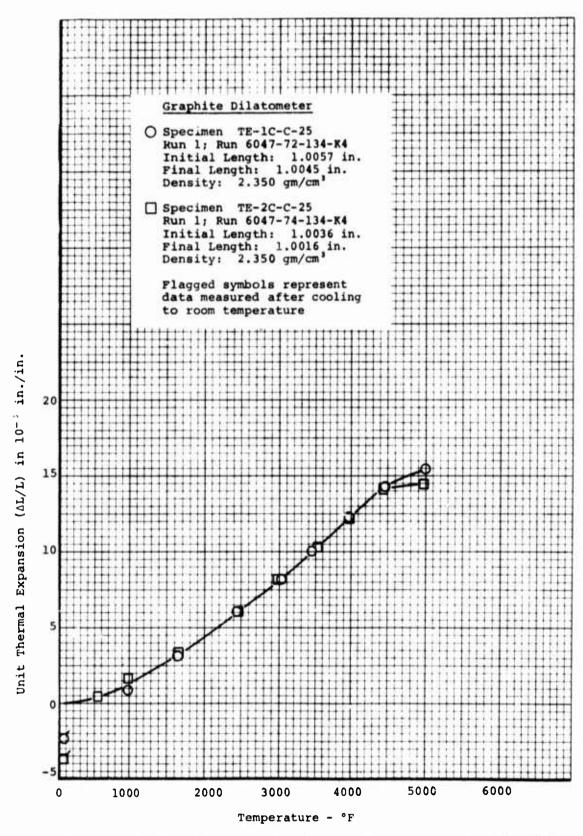


Figure 10. Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 27 Percent SiC in the 'C' Direction

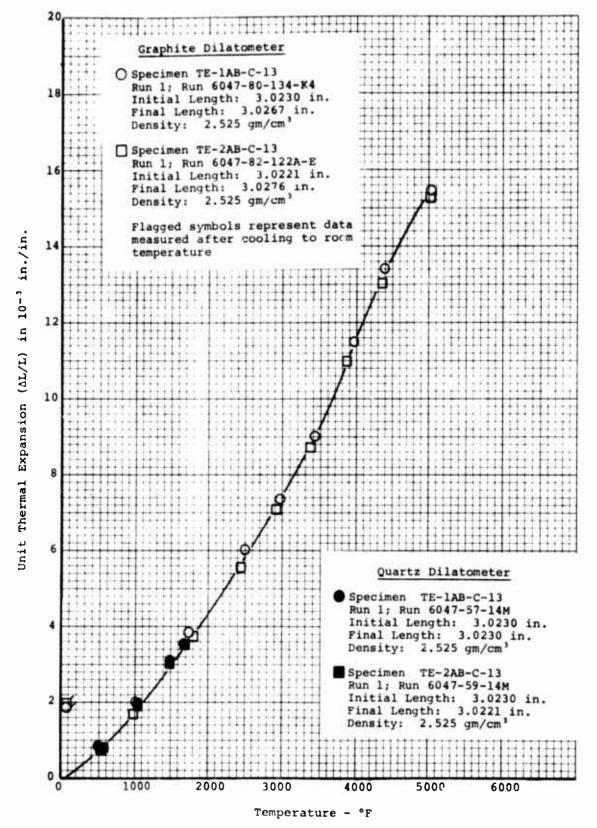


Figure 11. Thermal Expansion of Codeposited Pyrolytic Sleeve (Coupon Specimen) with 41 Percent SiC in the 'A-B' Direction

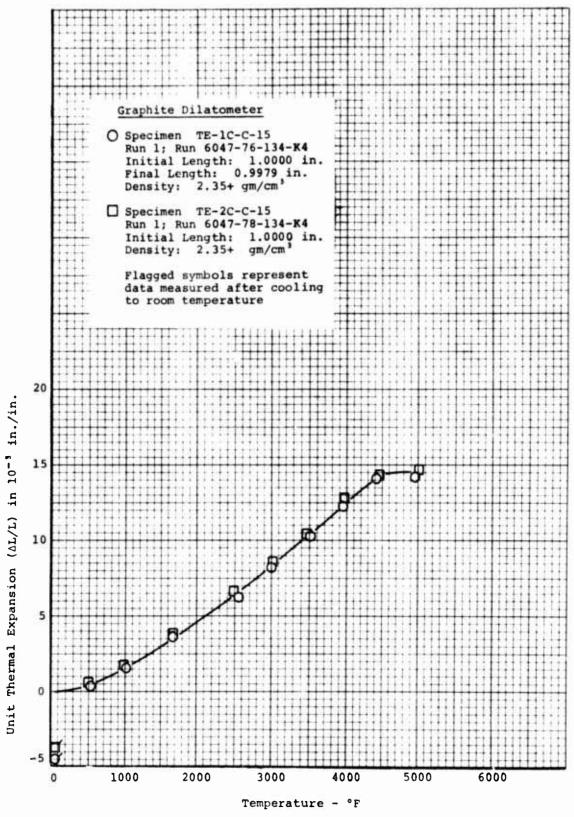


Figure 12. Thermal Expansion of Codeposited Pyrolytic Graphite Sleeve (Coupon Specimen) with 30 Percent SiC in the 'C' Direction

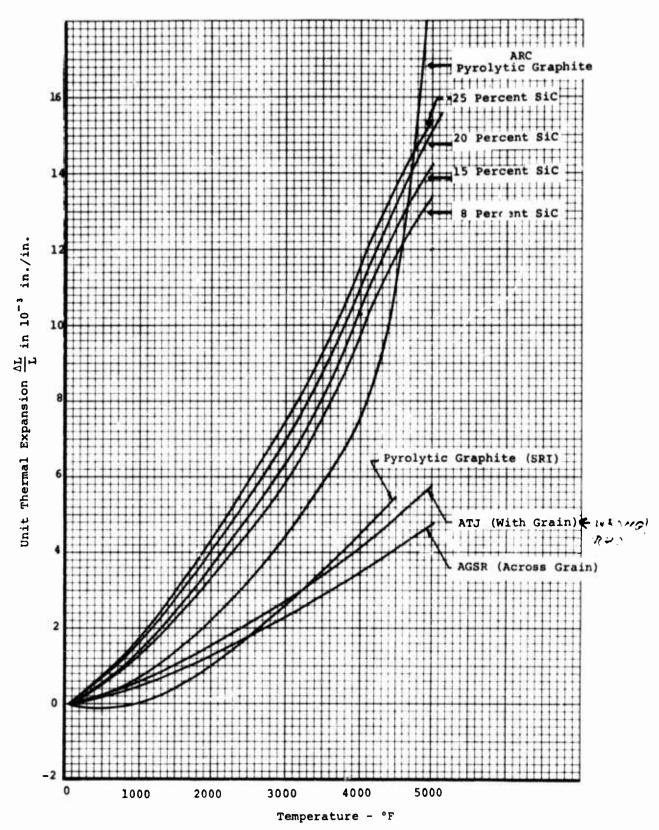


Figure 13. A Composite Plot of Thermal Expansion in the 'A-B' Direction

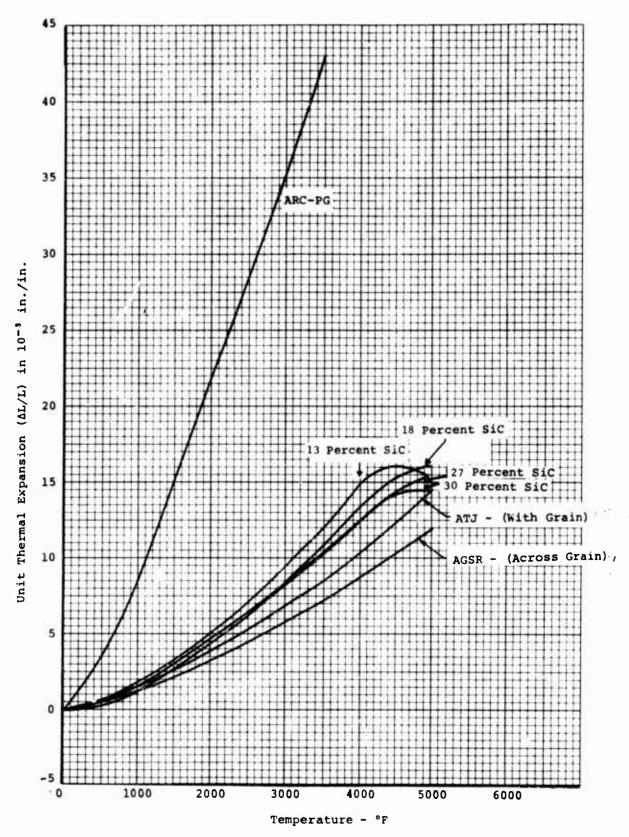
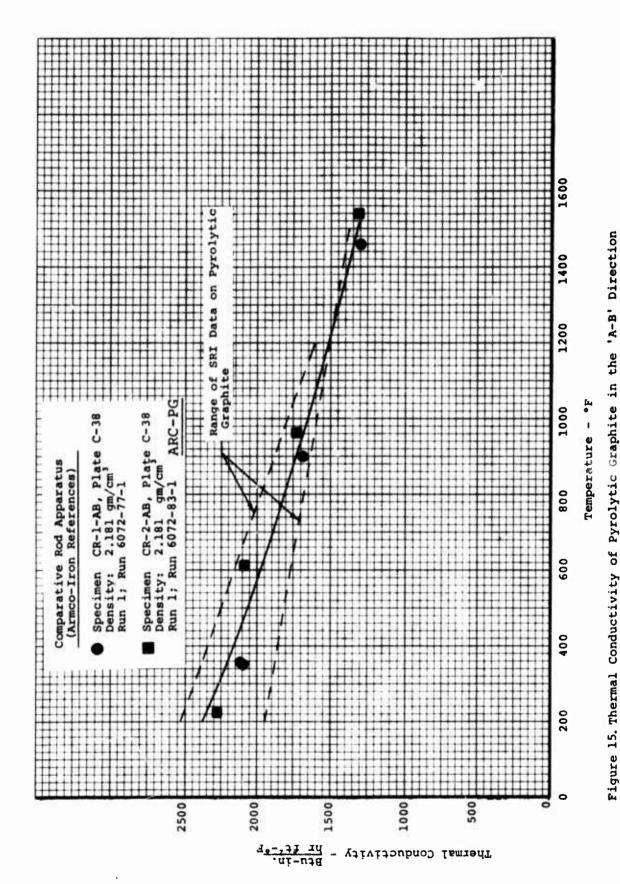


Figure 14. A Composite Plot of the Thermal Expansion in the 'C' Direction



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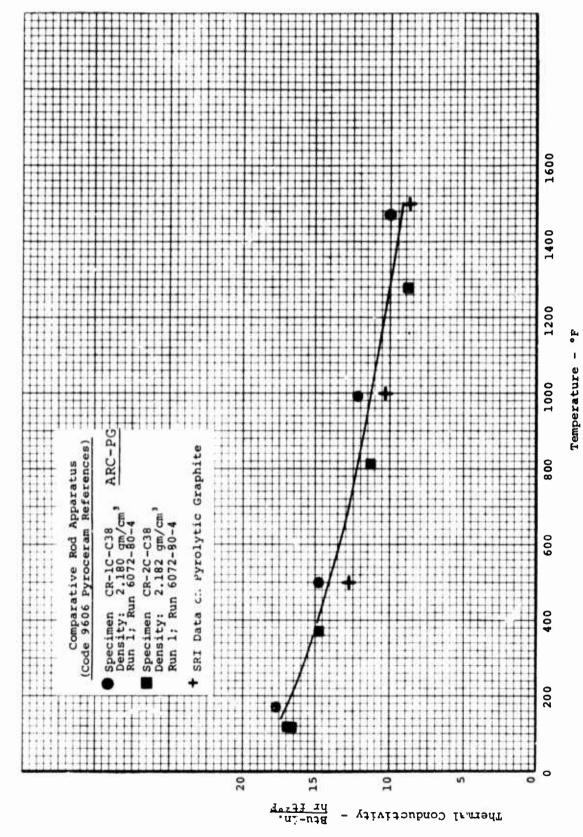
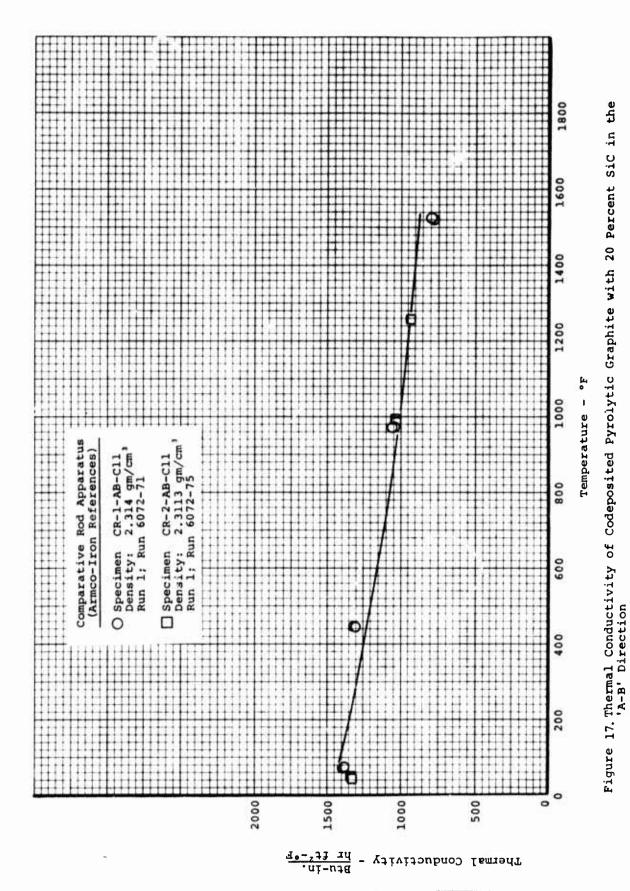
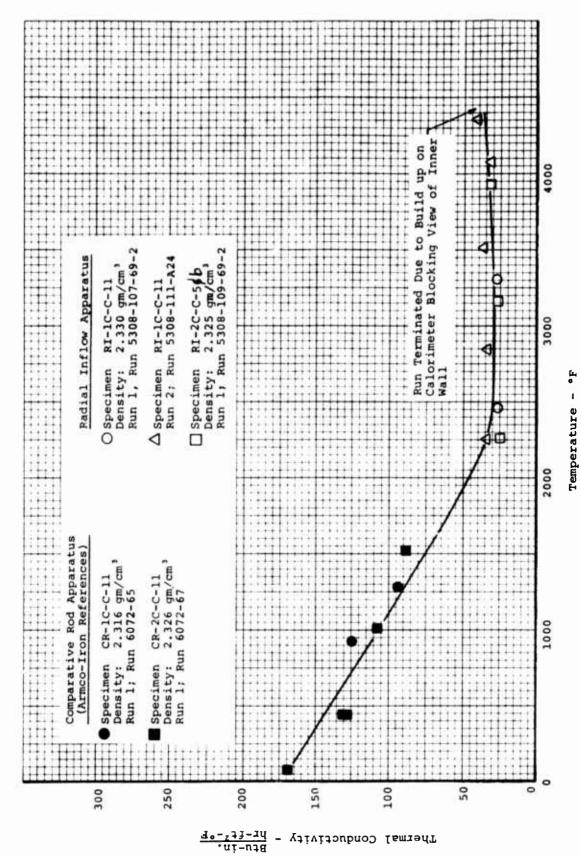


Figure 16. Thermal Conductivity of Pyrolytic Graphite in the 'C' Direction



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Thermal Conductivity of Codeposited Pyrolytic Graphite with 20 Percent SiC in the 'C' Direction Figure 18.

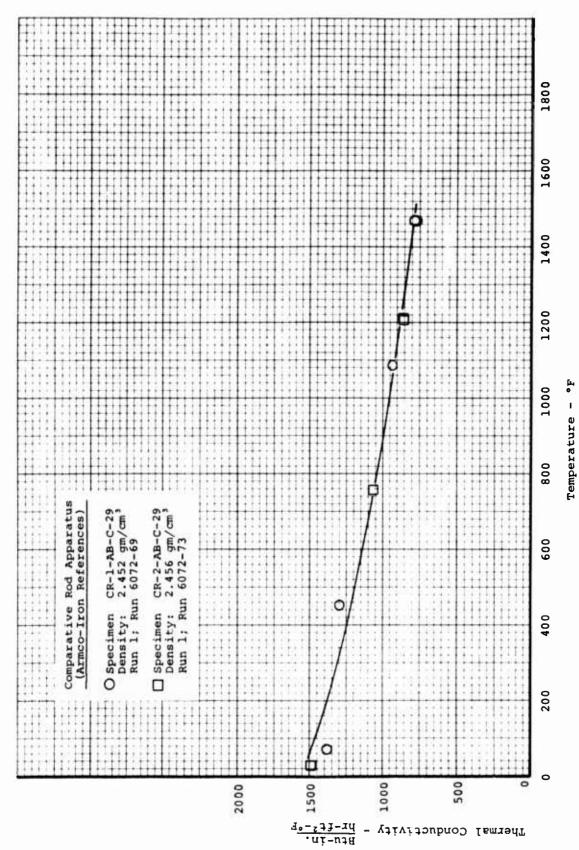


Figure 19. Thermal Conductivity of Codeposited Pyrolytic Graphite with 37 Percent SiC in the 'A-B' Direction

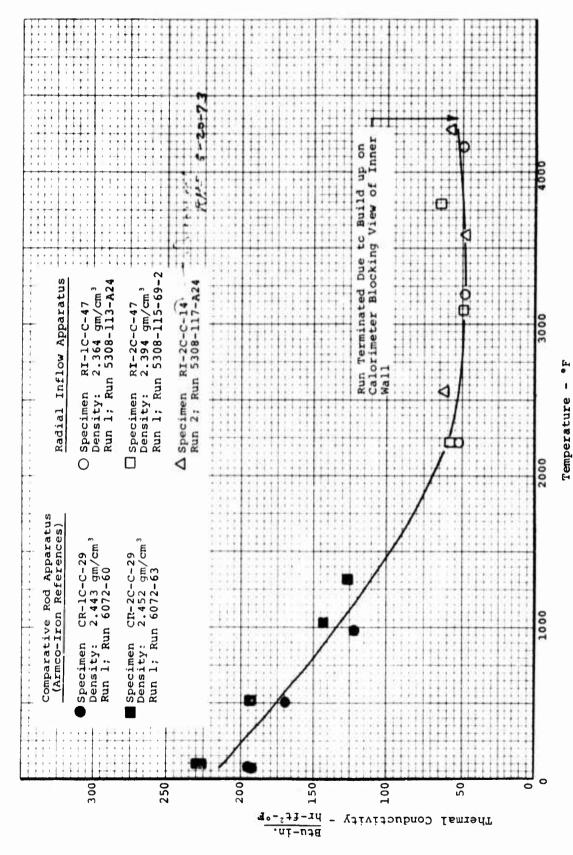
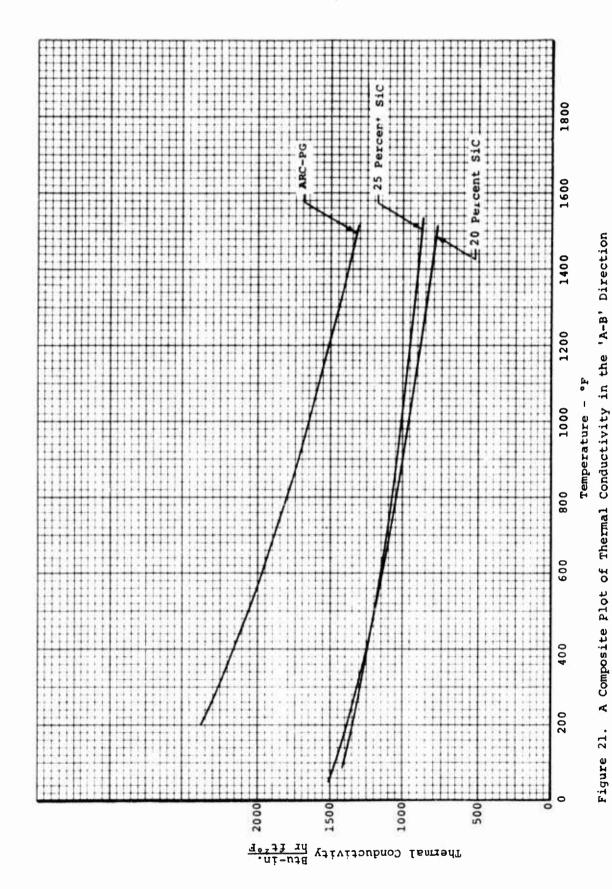
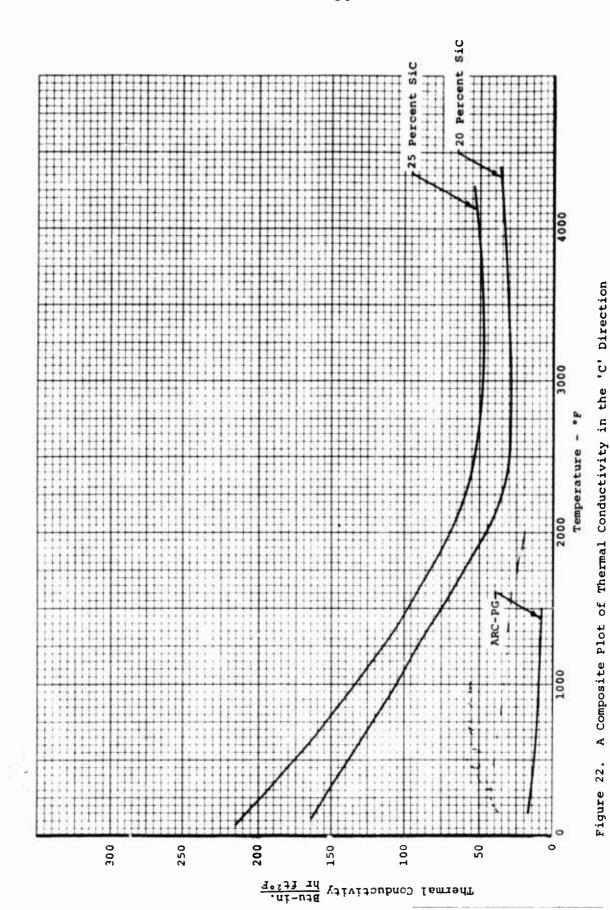
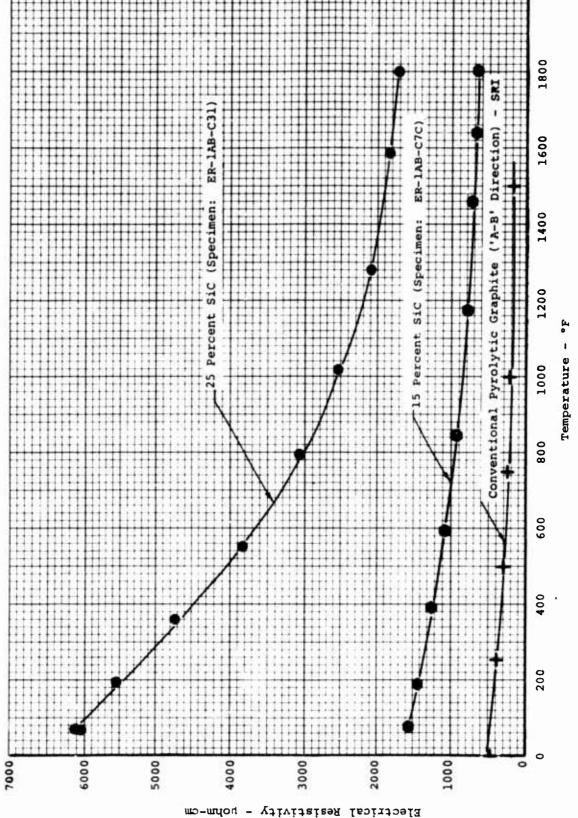


Figure 20. Thermal Conductivity of Codeposited Pyrolytic Graphite Sleeve with 37 Percent SiC in the 'C' Direction



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Electrical Resistivity of Codeposited Pyrolytic Graphite with 25 Percent and 15 Percent SiC in the 'A-B' Direction Figure 23.

Test	Material	Direction	Temperature °F
Thermal Expansion	ARC PG	'a-b' and 'c'	70 to 5000
	SiC/PG with 8% SiC	'a-b' and 'c'	70 to 5000
	SiC/PG with 15% SiC	'a-b' and 'c'	70 to 5000
	SiC/PG with 20% SiC	'a-b' and 'c'	70 to 5000
	SiC/PG with 25% SiC	'a-b' and 'c'	70 to 5000
Thermal Conductivity	ARC PG	'a-b' and 'c'	70 to 1700
Conductivity	SiC/PG with	'ab'	70 to 1700
	20% SiC	'c'	70 to 5000
	SiC/PG with	'ab'	70 to 1700
	25% SiC	'c'	70 to 5000
Electrical Resistivity	ARC PG	'ab'	70 to 1800
Resiscivity	SiC/PG with 15% SiC	'ab'	70 to 1800
	SiC/PG with 25% SiC	'ab'	70 to 1800

Table 2
Thermal Expansion of Pyrolytic Graphite in the 'A-B' Direction
Measured in Quartz Dilatometer

			Specimen Temperature -	perature - °F		Observed	Observed	Unit Elongation Corrected Correction for Specimen	Corrected
Specimen	Time	Top	Middle	Bottom	00000	Total Elongation	Unit Elongation	Dilatometer	Unit
Specimen: TE-1AB		Initial Length: Final Length:	h: 3.0010 3.0022	in. in.		Initial Weight: 2.7344 Final Weight: 2.7328	ght: 2.7344 t: 2.7328	4 gm 8 gm	10 in./in.
Run: 1 Run: 6047-51-20 Density: 2.195	11:00	73	73	73	73	0.00	0.00	0.00	0.00
	1:15	1000	1000	1000	1000	3.12	1.04	0.27	0.81
	2:30	1701	1700	1702	1701	3.79	1.26	0.50	1.76
Specimen: TE-2AB		Initial Lengt Final Length:	h: 3.0123 3.0125	in. in.		Initial Weight: Final Weight:	ght: 2.7835	e gm	
4	11:20	500	500	500	500	0.00	0.00	0.00	0.00
gm/cm³	1:55 2:20 7:40	1517	1521 1710 72	1525 1717 72	1521	3.45 4.22 -0.01	1.1.0	0.38	1.43

Table 3
Thermal Expansion of Pyrolytic Graphite in the 'A-B' Direction Measured in Graphite Dilatometer

Corrected Specimen Unit Elongation (10 ⁻³ in./in.)	0.00 0.19 1.62 1.62 2.85 4.06 10.61 10.21 1.67 1.67 1.67 1.67 1.67 1.67 1.67 1.6
Unit Correction for Dilatometer Motion (10-3 in. /in.)	0.00 0.25 0.25 0.25 0.00 0.00 0.00 0.00
Observed Unit Elongation (10 ⁻³ in./in.)	- 0.06 - 0.15 - 0.15 - 0.15 - 0.07 - 0.07 - 0.08 - 0.08 - 0.09 - 0.09 - 0.09 - 0.09 - 0.09 - 0.09 - 0.09 - 0.09 - 0.09 - 0.09
Observed Total Elongation (10 ⁻⁵ in.)	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Time	110.00 120.00 120.00 120.00 120.00 120.00 120.00 120.00 120.00 120.00 130.00
Temp.	438 1000 11614 2261 2821 3523 4009 4416 475 1033 1620 2415 3969 4374 5034 5034
Specimen and Run No.	Specimen: TE-1AB-C2 Run: 6047-67-122A-E Initial length: 3.0022 in. Final length: 2.7328 gm Linitial Weight: 2.7321 gm Density: 2.195 gm/cm³ Specimen: TE-2AB-C2 Run: 6047-69-122A-E Initial length: 3.0125 in. Final length: 3.0586 in. Initial weight: 2.7865 gm Final weight: 2.7852 gm Density: 2.195 gm/cm³

Table 4

Thermal Expansion of Pyrolytic Graphite in the 'A-B' Direction (Second Exposure)
Measured in Graphite Dilatometer

Corrected Specimen Unit Elongation (10-3 in. /in.)	0.00 0.22 1.66 33.98 3.98 6.04 1.00 1.00
Unit Correction for Dilatometer Motion (10-3 in. /in.)	0.00 0.65 1.68 3.08 6.16 7.30 10.30 0.00
Observed Unit Elongation (10 ⁻³ in./in.)	10000 100000 10000 10000 10000 10000 10000 10000 10000 10000 100000 10000 10000 10000 10000 10000 10000 10000 10000 100000 10000
Observed Total Elongation (10-3 in.)	1.00 1.00 1.00 1.00 1.00 1.00 1.00 1.00
Time	8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8
Temp.	44 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4
Specimen and Run No.	Specimen: TE-2AB-C2 Run: 6047-83-134-K4 Initial length: 3.0586 in. Final length: 3.0624 in. Initial weight: 2.7852 gm Final weight: 2.7852 gm Density: 2.195 gm/cm ³

Table 5
Thermal Expansion of Pyrolytic Graphite in the 'C' Direction
Measured in Graphite Dilatometer

Corrected Specimen Unit Elongation (10 ⁻³ in. /in.)	0.00 3.74 17.17 27.11 27.11 35.62 44.29 56.11 37.32 -26.61 17.20 37.32 17.20 37.32 17.20 37.32 -26.61 37.32 -26.61 37.32 -27.82 -27.82 -27.82 -27.82 -37.82 -30.93 -30.87
Unit Correction for Dilatometer Motion (10-3 in. /in.)	0.00 1.30 4.50 6.86 9.05 11.51 17.85 17.85 17.25 11.70 13.78 18.00
Observed Unit Elongation (10 ⁻³ in./in.)	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
Observed Total Elongation (10-3 in.)	20.00 12.70 12.70 26.62 32.85 38.66 19.51 12.55 12.55 39.29 39.29 39.29 39.29
Time	1100 1100
Temp.	10580 10533 10523 10525 1053 1053 1053 1053 1053 1053 1053 105
Specimen and Run No.	Specimen: TE-1C-C-14 Run: 6047-58-134-K4 Initial length: 1.0020 in. Final length: 0.9772 in. Initial weight: 2.2181 gm Charles and complementation of the comple

Table 6

Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 8 Percent SiC in the 'A-B' Direction (Measured in Quartz Dilatometer)

			Specimen Temperature	erature - *F		Observed	Observed	Unit Elongation Correction for	Sp
Specimen	Time	Top	Middle	Bottom	Average	Elongation 10- in.	Elongation 10- in./in.	Dilatometer Motion 10-1 in./in.	Unit Tongation 10- in./in.
Specimen: TE-1AB		Initial length: Final length:	h: 3.003 3.003	in. in.		Initial weight: Final weight:	ight: 3.2992 it: 3.2982	mg mg	4
Run: 1 Run: 6047-50-14M Density: 2.241 gm/cm³	11:00 12:35 1:15 2:00 2:30	73 500 1000 1495 1694	500 1000 1500 1700	73 500 1000 1503 1704	73 500 1000 1499 1699	0.00 1.34 3.49 5.98 6.97	0.00 0.45 1.16 1.99 0.32	0.00 0.22 0.22 0.30	0.00 0.57 1.38 2.26 -0.04
Specimen: TE-2AB		Initial length Final length:	h: 3.0000	in. in.		Initial weight: Final weight:	ght: 3.0665 ht: 3.0660	5 gm 50 gm	
Run: 6047-63-14M-	11:00	70 500 1000 1496	70 500 1000 1500	70 500 1000 1506	70 500 1000 1501	0.00 1.47 3.82 6.46	0.00 0.49 1.27 2.15	0.00 0.12 0.22 0.27	0.00
mo /mr	2:25	1694	1700	1709	1701 70	7.53	2.51	0.00	2.81

Table 7

Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 8 Percent SiC in the 'A-B' Direction Measured in Graphite Dilatometer

Corrected Specimen Unit Elongation (10° in. /in.)	0.00 0.54 1.43 2.42 7.93 11.63 11.63 13.51 13.51 13.51 13.53 13.23 13.23 3.10
Unit Correction for Dilatometer Motion (10-3 in. /in.)	0.00 10.928 10.00
Observed Unit Elongation (10 ⁻³ in./in.)	0.00 0.75 11.10 0.02 12.13 13.23 10.00 0.26 0.26 0.51 11.39 11.39 3.23 3.23 3.23 3.23 3.23 3.23 3.23
Observed Total Elongation (10-3 in.)	0.00 1.59 1.59 1.25 1.25 1.25 1.25 1.25 1.25 1.25 1.25
Time	11110098 888 112110009 89817517 11211000000000000000000000000000000000
Temp.	110473 10473 10
Specimen and Run No.	Specimen: TE-lAB-C- Run: 1 Run: 6047-71-122A-E Initial length: 3.0003 in. Final length: 3.0094 in. Initial weight: 2.9211 gm Density: 2.241 gm/cm ³ Specimen: TE-2AB-C- Run: 1 Run: 1 Run: 1 Run: 3.0000 in. Final length: 3.0005 in. Initial weight: 3.0095 in. Initial weight: 2.7183 gm Final weight: 2.7183 gm Density: 2.241 gm/cm ³

Table 8

Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 8 Percent SiC in the 'C' Direction Measured in Graphite Dilatometer

			Total	Unit	Unit Correction for	Corrected Specimen
Specimen and	Temp.		Elongation	Elongation	Dilatometer Motion	Unit Elongation
Run No.	٠ ټ	Time	(10 ⁻³ in.)	(10 ⁻³ in./in.)	(10 ⁻³ in. /in.)	(10 ⁻³ in. /in.)
Specimen: TE-1C-C-19-	30	0:	•	0	0	0
4	2	4	.2	7		. 0
Run: 1	œ	:2	9	9.0	5	00
Run: 6047-62-134-K4	63	0:0	0.6	9.0	'n	6
Initial length:	44	4:	2	0.5	(0
1.0050 in.	66	1:1	Τ.		. "	4
inal length:	55	2:1	0	9	9	2.6
0.9961 in.	0	2:5	٠.	5	3.6	5.1
Initial weight:	4496	1:30	0.21	0.21	15.80	16.01
2.2771 gm	่อ	0:	2.4	4	8.0	5.5
Final Weignt:	08		2	ω σ.	•	6.
Density: 2.241 gm/cm ³						
Specimen: TE-2C-C-19-	80	0:	0.	0	0	0
Ø	53	 	0.4	0.4	7	. 7
Run: 1	00	9:0	0.5	0.5	.5	6.
Run: 6047-64-134-K4	19	0:1	'n.	ů.	3	0
nitial length:	40	0:4	0.1	0	7	0
.ni 0000.1	2982	11:15	0.03	0.03	9.30	9
Final Length:	2	T:7	•	•	1.6	2.7
.ui 6686.0	5	2:4	φ.	ω.	9.	4.
Initial weight:	49	::	S.	5	5.8	6.3
2.2247 gm	07		3.0	3.0	8.0	4.9
Final weight:	80		ω.	10.8	0	0.8
2.0788 gm						
Density: 2.241 gm/cm ³						

Table 9
Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 15 Percent SiC in the 'A-B' Direction Measured in Quartz Dilatometer

SP	Unit Elongation 10 ⁻³ in./in.	1	0.00 0.62 1.55 2.62 3.08	0.00 0.66 1.55 2.70 2.96
Unit Elongation Correction for	Motion 10-3 in./in.	mg gm	0.00 0.10 0.27 0.50	0.00 0.12 0.22 0.30 0.00
ed	Elongation Elongation 10- in. 10- in./in.	ght: 3.7492 it: 3.7485	0.00 0.52 1.28 2.18 2.58	ght: 3.1616 0.00 0.54 1.33 2.42 2.66 -0.02
Observed	Elongation 10- in.	Initial weight: Final weight:	0.00 3.85 6.55 7.75	Initial weight: Final weight: 0.00 1.62 4.00 7.27 7.98 -0.05
	Average		72 500 1000 1500 1700	71 500 1000 1571 71
oerature - °F	Bottom	in. in.	72 500 1000 1503 1704	in. in. 71 500 1000 1575 1705
Specimen Temperature	Middle	th: 3.0003	72 506 1000 1500 1700	h: 3.0000 3.0000 71 500 1500 1571 1700 71
	Top	Initial leng Final length	72 500 1000 1498 1696	Initial length: Final length: 71 500 1000 1566 1694 71
	Time		11:00 12:40 1:20 2:05 2:30 7:30	10:30 12:25 1:05 1:05 2:10 8:15
	Specimen	Specimen: TE-lAB	Run: 1 Run: 6047-53-20 Density: 2.275 gm/cm ³	Specimen: TE-2AB Run: 1 Run: 6047-61-14M- 0 Density: 2.275 gm/cm ³

Table 10

Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 15 Percent SiC in the 'A-B' Direction (Measured in Graphite Dilatometer)

Corrected Specimen Unit Elongation (10 ⁻³ in. /in.)	0.00 1.56 2.85 6.45 10.65 12.87 14.36 1.54 3.18 8.12 12.88 14.24
Unit Correction for Dilatometer Motion (10-3 in. /in.)	0.00 11.90 11.90 0.00 0.00 0.00 0.00 0.0
Observed Unit Elongation (10 ⁻³ in./in.)	0.00 0.000 11.51 12.731 13.931 10.00
Observed Total Elongation (10-3 in.)	0.00 0.85 1.83 2.84 4.53 5.70 8.21 11.50 14.90 11.91 3.37 4.85 6.25 7.90 14.22 14.22
Time	1100 6 6 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Temp.	7 8 1 1052 1 1658 2 2523 3 3018 3 3018 3 3018 4 4 0 2 9 1 1 0 2 9 1 1 0 2 9 2 5 3 0 3 3 4 4 7 4 3 6 7 7 8 7 8 7 8 8 1 1 9 7 8 8 1 1 9 7 8 7 8 8 1 8 7 8 8 1 8 7 8 8 1 8 7 8 8 1 8 8 1 8 8 1 8 1 8 1 8 1 8 1 8 1
Specimen and Run No.	Specimen: TE-lAB-C-7 Run: 1 Run: 6047-75-122A-E Initial length: 3.0006 in. Final length: 3.7485 gm Final weight: 3.7485 gm Final weight: 7-D Run: 1 Run: 6047-77-122-A-E Initial length: 3.0000 in. Final length: 3.1005 in. Initial weight: 3.1611 gm Final weight: 2.275 gm/cm ³ Density: 2.275 gm/cm ³

Table 11

Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 15 Percent SiC in the 'C' Direction (Measured in Graphite Dilatometer)

Corrected Specimen Unit Elongation (10 ⁻³ in. /in.)	0.00 1.21 10.92 10.92 113.41 15.35 1.74 11.29 13.86 15.70 16.26
Unit Correction for Dilatometer Motion (10-3 in. /in.)	0.00 1.21 2.60 7.16 13.50 15.95 17.90 0.00 1.28 4.50 9.71 13.78 18.10 0.00
Observed Unit Elongation (10 ⁻³ in./in.)	0.00 1.1.36 1.39 1.39 1.39 1.39 1.39 1.39 1.39 1.39
Observed Total Elongation (10-3 in.)	0.00 -1.25 -1.30 -1.30 -1.30 -1.07 -1.07 -1.096 -1.07
Time	8:45 9:15 10:20 10:50 11:20 11:20 12:50 11:25 7:35 11:25 11:
Temp.	1005 1006 10006 30392 30992 45993 10057 10057 24523 30083 480683 788 788 788 788 788 788 788 788 788 7
Specimen and Run No.	Specimen: TE-1C-C-7A Run: 1 Run: 6047-66-134-K4 Initial length: 1.0755 in. Final length: 2.4275 gm Final weight: 2.2688 gm Density: 2.275 gm/cm³ Specimen: TE-2C-C-7A Run: 1 Run: 6047-70-134-K-4 Initial length: 0.9929 in. Final length: 0.9929 in. Initial weight: 2.2623 gm Final weight: 2.2623 gm Density: 2.275 gm/cm³ Density: 2.275 gm/cm³

Table 12

Thermal Expansion of Codeposited Pyrolytic Graphite (Coupon Specimen) with 20 Percent SiC in the 'A-B' Direction Measured in Quartz Dilatometer

			Specimen Temperature	erature - *F		Observed	Observed	Unit Elongation Corrected Correction for Specimen	Corrected Specimen
Specimen	Time	Top	Middle	Bottom	Average	Elongation Elongation 10-1 in. 10-1 in. /in	Unit Elongation 10-1in./in.	Dilatometer Motion 10-3 in./in.	Unit Elongation 10- in./in.
Specimen: TS-2AB		Initial length: Final length:	angth: 3.0020 in. gth: 3.0020 in.	in.		Initial weight: Final weight:	ight: 4.9111 pht: 4.7215	11 gm 15 gm	
Run: 1 Run: 6047-65-14M- Q Density: 2.350	10:00	74	74	74	74 598	0.00	00.0	0.00	0.00
gm/cm³	1:10 1:50 2:15 7:30	1006 1565 1694 74	1000 1569 1700	1000 1577 1709 74	1000 1570 1701 74	4.66 8.19 9.04	1.55 2.73 3.01 -0.01	0.22 0.28 0.31 0.00	3.01 3.32 -0.01
Specimen: TE-1AB		Initial length: Final length:	ength: 3.0024 gth: 3.0023	in.		Initial weight: Final weight:	ight: 4.9048 ht: 4.9037	18 gm 37 gm	
-C-17 Run: 1 Run: 6047-65-14M-	11:00	72 500 500	72 500 1000	72 500 1000	72 500 1000	0.00 1.96 4.90	0.00	0.00	0.00
Density: 2.350 gm/cm³	7:30 7:30	1496 1693 76	1500 1700 176	1505 1708 76	1500	8.11 9.43 -0.36	2.70 3.14 -0.12	0.27 0.30 0.00	2.97 3.44 -0.12

Table 13

Thermal Expansion of Codeposited Pyrolytic Graphite (Coupon Specimen) with 20 Fercent SiC in the 'A-B' Direction Measured in Graphite Dilatometer

Corrected Specimen Unit Elongation (10 ⁻³ in. /in.)	0.00 0.68 1.83 3.68 5.72 7.33 8.62 11.18 13.76 15.58 3.50 0.00 0.69 1.85 3.30 6.92 8.75 11.37 13.30 6.65 6.65
Unit Correction for Dilatometer Motion (10 ⁻³ in. /in.)	for 5000°F
Observed Unit Elongation (10 ⁻³ in./in.)	78 8:35 0.00 0.00 0.39 78 10:10 1.17 0.39 78 10:10 4.74 1.58 78 10:10 4.74 1.58 78 11:15 8.06 2.68 70 11:45 9.80 3.26 712:45 11:45 9.80 3.26 712:45 11:45 13.72 4.57 567 12:45 16.85 5.61 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 77 8:45 0.00 0.00 78 11:30 0.00 78 11:30 0.00 79 11:30 0.00 70 19:97 0.66 70 19:97 0.66 8 11:30 15:52 5.17 70 8:00 19:97 0.66 8 11:30 0.00 0.00 while going exposure temperature not known.
Observed Total Elongation (10-3 in.)	0.00 1.17 2.79 4.74 6.53 8.06 9.80 13.72 16.85 17.97 10.50 1.12 2.54 3.95 5.56 6.72 6.72 9.17 13.17 13.17 13.17 19.97
Time	78 8:35 82 9:10 35 9:40 81 10:10 36 10:40 78 11:15 02 11:45 64 12:15 67 12:45 77 8:45 77 8:45 77 8:45 10:30 68 11:30 68 11:30
Temp.	78 482 1035 1035 1781 2536 3068 34567 4567 527 1106 1760 2575 3068 3543 4095 4095 4095
Specimen and Run No.	Specimen: TE-lAB-C-17 Run: 1 Run: 6047-79-122A-E Initial length: 3.0023 in. Final length: 4.9037 gm Final weight: 3.6071 gm Density: 2.350 gm/cm³ Specimen: TE-2AB-C-17 Run: 1 Run: 6047-81-122A-E Initial length: 3.0015 in. Final weight: 3.0215 in. Initial weight: 4.7215 gm Final weight: 4.7215 gm Final weight: 5.330 gm/cm³ Density: 2.350 gm/cm³

Table 14

Thermal Expansion of Codeposited Pyrolytic Graphite Plate with 20 Percent SiC in the 'C' Direction Measured in Graphite Dilatometer)

Corrected Specimen Unit Elongation (10 ⁻³ in. /in.)	0.00 3.15 6.07 8.21 10.04 12.39 14.37 15.49 0.00 0.49 0.00 1.62 1.63 1.64 1.
Unit Correction for Dilatometer Motion (10 ⁻³ in. /in.)	0.00 2.48 4.55 7.30 11.16 13.37 15.52 17.90 0.00 1.18 9.34 11.48 113.38 17.86 0.00
Observed Unit Elongation (10-3in./in.)	10.000 11.123 11.123 11.123 12.1133 13.38 13.38
Observed Total Elongation (10-3 in.)	0 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Time	8: 10: 10: 10: 10: 10: 10: 10: 10
Temp.	1628 1628 1628 30448 34948 1669 1669 177 1899 1899 1899 1899 1899 1899 1899
Specimen and Run No.	Specimen: TE-1C-C-25 Run: 1 Run: 6047-72-134-K4 Initial length: 1.0057 in. Final length: 2.4371 gm Final weight: 2.1854 gm Density: 2.350 gm/cm³ Specimen: TE-2C-C-25 Run: 6047-74-134-K4 Initial length: 1.0036 in. Final length: 2.4250 gm Final weight: 2.4250 gm Final weight: 2.4250 gm Final weight: 2.4250 gm Final weight: 2.4250 gm Density: 2.350 gm/cm³

Table 15

Thermal Expansion of Codeposited Pyrolytic Graphite (Coupon Specimen) with 25 Percent SiC in the 'A-B' Direction (Measured in Quartz Dilatometer)

			Specimen Temperature	perature - °F		Observed	Observed	Unit Elongation Corrected	Corrected Specimen
Specimen	Time	Top	Middle	Bottom	Average	Elongation Elongation 10-1 in./in	Elongation 10- in./in.	Motion 10 ⁻³ in./in.	Elongation 10-1 in./in.
Specimen: TE-2AB		Initial length: Final length:	ength: 3.0230 gth: 3.0221	in. I in.		Initial weight: Final weight:	ight: 5.0520 ht: 5.0510	.20 gm .10 gm	
4	9:45	500	500	500	500	0.00	0.00	0.00	0.00
Density: 2.525 gm/cm ³	1:25 1:25 1:50 8:30	1022 1496 1687 72	1622 1500 1694 72	1505 1701 72	1500 1694 72	9.83 -0.22	3.27	0.30 0.00	3.06
Specimen: TE-1AB		Initial length: Final length:	ength: 3.0230 gth: 3.0230	0 in.		Initial weight: Final weight:	ight: 5.0235 nt: 5.0229	15 gm 19 gm	
Run: 1 Run: 6047-57-14M-	8:45 9:20	75 500 1037	75 500 1039	75 500 1039	75 500 1038	0.00 2.08 5.36	0.00 0.69 1.77	0.00 0.12 0.23	0.00 0.81 2.00
Density: 2.525 gm/cm³	10:30 11:00 8:20	1496 1630 75	1500 1700 75	1705	1500 1699 75	8.62 9.92 -0.25	2.85 3.28 -0.08	0.27	3.12 3.58 -0.08

Table 16

Thermal Expansion of Codeposited Pyrolytic Graphite (Coupon Specimen) with 25 Percent SiC in the 'A-B' Direction Measured in Graphite Dilatometer)

			Observed	Observed	Init Correction for	- C
Specimen and Run No.	Temp.	Time	Elongation (10-3 in)	Elongation (10-3in /in)	Dilatometer Motion	Unit Elongation
	•		,::::	/= (,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,		(10 m. / m.)
Specimen: TE-lAB-C-13	7	.5	0.	0	0	0.
Run: 1	49	.3	9.	7.	9	œ
Run: 6047-80-134-K4	00	0:0	7	4	9.	°.
Initial length:	70	0:3	7	7	1	ω.
3.0230 in.	50	1:0	٣.	0.	6	°.
Final length:	96	1:3	6	.2	٦.	۳.
3.0267 in.	3412	0:	7	.7	E.	9.0
Initial weight:	95	2:3	σ.	9.	8.8	'n
5.0229 gm	39	0:			٦.	3.4
Final weight:	5034	1:30	9.85	3.26	12.24	15.50
3.9385 gm	73	(;)		φ.	۰.	φ.
Density: 2.525 gm/cm ³						
Specimen: TE-2AB-C-13	7	<u>ب</u>	0	0	0	0.
Run: 1	52	0:	۳.	4	4	7.
Run: 6047-82-122A-E	98	9:3	.5	œ	00	9
Initial length:	79	0:0	۰.	9	4	
3.0221 in.	42	0:3	6.	7	٣.	ທຸ
Final length:	91	1:0	8.3		۳.	٠.
3.0276 in.	3371	11:30	10.33	3.42	5.32	8.74
Initial weight:	88	2:5	3.5	'n	4	S
5.0510 gm	36	2:5	5.2	۳.	9.	٠,
Final weight:	8	<u>ښ</u>	ં. જ	٥.	7	2
3.4192 gm	7.8	. :	6.	σ.	0.	5
Density: 2.525 gm/cm ³						

Table 17

Thermal Expansion of Codeposited Pyrolytic Graphite (Coupon Specimen) with 25 Percent SiC in the 'C' Direction (Measured in Graphite Dilatometer)

Specimen and Run No.	Temp.	Time	Observed Total Elongation (10-3 in.)	Observed Unit Elongation (10 ⁻³ in./in.)	Unit Correction for Dilatometer Motion (10 ⁻³ in. /in.)	Corrected Specimen Unit Elongation (10 ⁻³ in. /in.)
Specimen: TE-1C-C-15 Run: 1 Run: 6047-76-134-K4 Initial length: 1.0000 in. Final length: 0.9979 in. Initial weight: 2.4221 gm Final weight: 2.1739 gm Density: 2.35 gm/cm³	74 517 1015 1678 2528 3508 3963 4415 4983	8:20 9:00 9:40 10:15 10:55 11:30 12:45 1:15 7:35	0.00 -0.70 -1.05 -1.13 -1.120 -1.20 -1.41	0.00 -0.70 -1.05 -1.13 -1.12 -1.20 -1.20 -1.41	0.00 1.19 2.60 4.70 7.55 9.38 11.48 13.35 17.90 0.00	0.00 0.49 1.55 3.57 8.26 10.28 14.01 14.01
Specimen: TE-2C-C-15 Run: 1 Run: 6047-78-134-K4 Initial length: 1.0000 in. Final length: 2.4320 gm Final weight: 2.4320 gm Final weight: 2.2511 gm Density: 2.35+ gm/cm ³	78 491 1685 1685 2480 3008 3463 44476 5014	8:35 9:10 10:10 11:15 11:15 11:15 1:15 8:00	0.00 -0.46 -0.64 -0.76 -0.79 -0.76 -1.41	0.00 -0.64 -0.76 -0.79 -0.79 -0.79 -1.41 -1.41	0.00 1.10 2.49 4.71 7.43 9.43 11.24 13.45 18.00	0.00 0.64 1.85 3.95 8.61 10.48 14.34 14.34
	Final len specimen	length men to	of specim stick to	of	obtained due to SiC depo of dilatometer	deposites causing

Table 18

Thermal Conductivity of Pyrolytic Graphite in the 'A-B' Direction Measured in Comparative Rod Apparatus with Armco-Iron References

AT through Upper Reference AT2			59.25	146.6	186.4	186.6	<u> </u>		27.10	140.2	140.2	178.8
Thermal Conductivity of Upper Reference R ₂ Rtu-in./hr ft²*F	8.5005 gm 8.4633 cm		404 409	275	193	193	11.0282 gm 10.9848 gm		451 451	345	343	196
Mean Tempera- ture of Upper Reference	ټ		435	1055	1631	1633	; ;		258	755	758	1706
AT through Lower Reference AT1	Initial Weigh Final Weight:		53.13	125.9	125.6	157.3	Initial Weigh Final Weight:		25.24	112.5	113.1	171.0
Thermal Conductivity of Lower Reference X ₁ Stu-in./hr ft²°F	1.4998 in. 1.5000 in.		440	331	331 234	234	1.5731 in.		470	393	392	222
Hean Tempera- ture of through Lower Specimen Reference	1		302	296	1299	1300			191	483	486	823 1373
ΔT through Specimen	Initial Thickness: Final Thickness:		18.80	40.78	41.00	46.5	Initial Thickness: Final Thickness:		9.34	39.15	39.00	44.94
Thermal Conductivit of Specimen Kg Btu-in./hr ft ² °F	in.		2103	1675	1667	1305	50 in.		2274	2085	2095	1724
Mean Temperature of Specimen	1 = 1 = 0.750 1 = 1:2500 in.		364	668	899	1458	$1 = 1 = 0.7$ $1_{s}^{1} = 1.3231 \text{ in}$		225	612	615	961 1541
Specimen and Time	Specimen: CR-1-AB-C-38	gm/cm ³ Run: 6072-77-1	11:45	2:55	3:30	7:30	imen: CR-2-	Run: 6072-83-1 Density: 2.181 gm/cm	9:20	1:40	2:00	3:00

All measurements made with helium purge. Thermal conductivity $\left(k_S\right)$ of specimen calculated from following equation Notes: 1.

$$k_{S} = \begin{bmatrix} k_{1} \Delta T_{1} \\ l_{1} \end{bmatrix} + \frac{k_{2} \Delta T_{2}}{l_{2}} \end{bmatrix} \frac{l_{S}}{2\Delta T_{S}}$$

k = thermal conductivity

 ΔT = temperature drop over gage length 1 = gage length

and subscripts 1, 2, and s refer to lower reference, upper reference, and specimen, respectively.

Table 19

Thermal Conductivity of Pyrolytic Graphite in the 'C' Direction Measured in Comparative Rod Apparatus with Code 9606 Pyroceram References

						
AT through Upper Reference AT:		30.1	119.2		28.0	101.7
Thermal Conductivity of Upper Reference K ₂ Btu-in./hr ft ² *F	75 gm 93 gm	26.0 26.0 23.38	21.4 19.82	318 gm 279 gm	26.62	22.0 22.0 20.4
Mean Tempera- ture.of Upper Reference	ight: 4.4575 ht: 4.4493	192 192 573	1087 1578	aight: 3.9318 jht: 3.9279	142	435 899 902 1374
ΔT through Lower Reference ΔT °F1	Initial Weight: Final Weight:	26.62 28.0 75.7	100.4	Initial Weight: Final Weight:	28.6	72.3 89.3 92.3
Thermal Conductivity of Lower Reference K ₁ Btu-in./hr ft ² °F	0.1585 in. 0.1583 in.	28.0 26.6 24.05	22.05 8.72	0.1398 in. 0.1396 in.	27.30	24.8 22.7 22.7 21.1
Hean Tempera- ture of through Lower Specimen Reference *F	Initial Thickness: Final Thickness:	142 142 435	902	Initial Thickness: Final Thickness:	92 92	308 729 722 1188
	Initial Thickness: Final Thickness:	9.16 9.05 26.61	38.6	 Initial Thicknes Final Thickness:	8.60	22.8 35.0 44.0
Thermal Conductivity of Specimen Ks Btu-in./hr ft²°F	750 in. in.	17.6	12.32	 = 0.750 in. 1398 in.	16.6 16.8	14.78 11.35 11.25 8.72
Mean Temperature of Specimen	1 = 1 = 0.750 in. 1s = 0.1585 in.	168 168 504	990	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	118	373 809 810 1276
Specimen and Time	Specimen: CR- 1C-C-38 Run: 1 Run: 6072-80-4 Density: 2,180	8:15 16:00 3:20	1:00 2:00 5:20	Specimen: CR- 2C Plate C-38	Run: 6072-80-4 Fun: 6072-80-4 Density: 2,182s 8:15 gm/cm 10:00	3:20 1:00 2:00 5:20

Notes: 1. All measurements made with helium purge. 2. Thermal conductivity $(k_{\rm S})$ of specimen calculated from following equation

$$k_S = \left[\frac{k_1 \ \Delta T_1}{l_1} \ + \frac{k_2 \ \Delta T_2}{l_2} \right] \frac{l_S}{2\Delta T_S}$$

k = thermal conductivity ΔT = temperature drop over gage length

l = gage length

and subscripts 1, 2, and s refer to lower reference, upper reference, and specimen, respectively.

Table 29

Thermal Conductivity of Codeposited Pyrolytic Graphite with 20 Percent SiC in the 'A-B' Direction (Measured in Comparative Rod Apparatus with Armco Iron References)

4 0			
ΔT through Upper Reference ΔT ₂		69.34 69.33 79.58 79.63 132.60 136.70 98.15	86.75 88.00 220.19 220.10 276.00
Thermal Conductivity of Upper Reference R ₁ R ₂	6.7184 gm 6.7110 gm	4884 3889 268 268 196	6.7080 gm 6.6983 gm 485 483 254 254
Mean Tempera- ture of Upper Reference			
ΔT through Lower Reference ΔT °F¹	Initial Weight: Final Weight:	63.85 63.75 72.85 72.73 112.92 111.00 85.46	Initial Weight: Final Weight: 80.50 144 82.00 154 176.17 1200 176.40 1202
Thermal Conductivity of Lower Reference R1 Btu-in./hr ft²eF	0.9010 in. 0.9010 in.	525 527 423 423 313 312 211	0.9008 in. 0.9008 in. 552 548 325 325 384
Mean Tempera- ture of through Lower Specimen Reference	Initial Thickness: Final Thickness:	11 10 371 373 869 870 1443	Initial Thickness: Final Thickness: 26.06 -62 26.25 -59 43.43 808 45.70 1021
	Initial Final Th	19.39 18.67 18.67 27.12 27.17 18.60	Initial Final Th 26.06 26.25 43.43 45.70
Thermal Conductivity of Specimen Ks Btu-in./hr ft²ººF	= 0.750 in.	1384 1392 1323 1362 1040 799	.750 in. in. 1328 1332 1044 1043
Mean Temperature of Specimen	1 = 1 = 0.75 13 = 0.600 in.	77 77 444 445 975 976 1522	1 = 1 = 0.750 1s = 0.600 in. 45 49 990 1258
Spr inen and Time	Specimen: CR-1 AB-C-11 Run: 6072-71 Density: 2.314	10:30 11:00 3:40 4:05 10:45 2:00 2:30	Specimen: CR-2A Run: 6072-75 Density: 2.3113 gm/cm ³ 10:00 10:30 12:50 2:00

Notes: 1. All measurements made with helium purge. 2. Thermal conductivity $(k_{\rm g})$ of specimen calculated from following equation

$$k_S = \left\lceil \frac{k_1 \ \Delta T_1}{l_1} + \frac{k_2 \ \Delta T_2}{l_2} \right\rceil \frac{l_S}{2\Delta T_S}$$

1 = gage length

and subscripts 1, 2, and s refer to lower reference, upper reference, and specimen, respectively.

Thermal Conductivity of Codeposited Pyrolytic Graphite with 20 Percent SiC in the 'C' Direction (Measured in Comparative Rod Apparatus with Type 316 Stainless Steel References)

through Upper Reference		149.35 150.57 109.20 109.05 140.22 140.40	118.25	78.67 78.7 79.5
Thermal Conductivity of Upper Reference Raference Raference	79 gm 73 gm	100.7 101.0 120.8 160.8 186.0	E 5	123 123 159 186
Mean Tempera- ture of Upper Reference	ight: 0.9379 ht: 0.9373	206 209 209 556 562 1179 1178	1678 ight: 0.9325 ht: 0.9320	557 557 1141 1669
through Lower Reference	Initial Weight: Final Weight:	177.25 178.90 120.60 120.68 155.66 155.55	127.08 16 Initial Weight: Final Weight:	83.9 83.9 81.8 107.1
Thermal Conductivity of Lower Reference R ₁ R ₁	0.1254 in. 0.1254 in.	80.0 80.0 108.0 140.0 140.0	170.1 0.1255 in. 0.1255 in.	110 111 145 170
Drrected Tempera- in ture of through Lower Specimen Reference	Initial Thickness: Final Thickness:	1 1 E E E E E E E E E E E E E E E E E E	40.62 1358 Initial Thickness: Final Thickness:	375 376 924 1348
	Initial Thickness:	14.49 17.10 16.81 16.81 34.12 40.75	40.62 1358 Initial Thickness:	13.13 13.10 24.54 48.62
Thermal Conductivity of Specimen Ks Btu-in./hr ft²9	= 0.750 in. 254 in.	169 170 128 132 109 89	90 750 in. In.	181 182 125 94
Mean Temperature of Specimen	1 = 1 = 0.750 1s = 0.1254 in.	62 64 443 444 1010 1519	$\begin{vmatrix} 1521 \\ 1 & = 1 \\ 1 & = 0.750 \\ 1 & = 0.1255 \text{ in.} \end{vmatrix}$	472 472 1034 1519
Specimen and Time	Specimen: CR-2C-Run: 1 -C-11 Run: 6072-67 Density: 2.326 gm/cm ³	8:50 9:15 2:40 3:30 12:45 12:45	ime	Aun: 1 Run: 672-65 Density: 2.316 3:10 3:25 1:00 3:05

$$k_S = \left[\frac{k_1 \Delta T_1}{l_1} + \frac{k_2 \Delta T_2}{l_2}\right] \frac{l_S}{2\Delta T_S}$$

k = thermal conductivity $\Delta T = temperature drop over gage length$

| = gage length

and subscripts 1, 2, and s refer to lower reference, upper reference, and specimen, respectively.

*Temperature gradient corrected for grafoil at interfaces from known in-house thermal conductivity of grafoil.

Notes: 1. All measurements made with helium purge. 2. Thermal conductivity $(k_{\rm S})$ of specimen calculated from following equation

Table 22 The Thermal Conductivity of Codeposited Pyrolytic Graphite Sleeve with 20 Percent SiC in the 'C' Direction (Measured in Radial Inflow Apparatus Using an Optical Technique)

Time	Outer Surface Tempera- ture PG-SiC °F	Inner Wall Tempera- ture PG-SiC °F	Specimen AT PG-SiC °F	Heat to Calorimeter Btu hr	Specimen Mean Temperature °F	Thermal Conductivity Btu-in. hr ft ²⁻⁶ F
Run: 1	Sleevo: R 08-107-69-2 2.330 gm/		R R		adius 2.000 i dius 1.7190	
11:00	2478	2430	48	181	2454	26.2
1:00	3366	3238	128	509	3302	27.6
2:00	Run term of inner	inated due	to build	-up on calori	meter blocking	g optical view
Specimen Run: 2 Run: 53	: RI-1C-C- 08-111-A24	11				
10:30	2277	2236	41	206	2256	34.8
12:15	2887	2800	87	426	2844	34.0
1:15	3591	3443	148	791	3517	37.1
2:15	4206	3963	243	1141	4085	32.6
3:00	4484	4222	262	1562	4353	41.3
3:45	Run term of inner		to build-	up on calori	meter blocking	g optical view
Run: 1 Run: 53	Sleeve: R 08-109-69-2 2.325 gm/				adius 2.000 in dius 1.7150 :	
9:30	2277	2233	44	152	2255	24.3
11:15	3221	3101	120	450	3161	26.4
12:45	4047	3816	231	1040	3932	31.7
1:20	Run term of inner	inated due	to build-	up on calori	meter blocking	optical view

The thermal conductivity was calculated from the Equation:

$$K = \frac{2n (R_2/R_1)}{2\pi L} \cdot \frac{Q}{\Delta T}$$

where

K = thermal conductivity

Q = heat to calorimeter ΔT = temperature difference between outside and inside radii

L = gage length of calorimeter 0.500 in.

Notes:

 $^{^{1}\}mathrm{All}$ measurements made in 1 atm helium purge after evacuating and back filling furnace twice with helium.

Table 23

Thermal Conductivity of Codeposited Pyrolytic Graphite with 25 Percent SiC in the A-B' Direction Measured in Comparative Rod Apparatus with Armco-Iron References)

through Upper Feference AT2		36.47 36.53 50.35 50.37 113.57 149.02		64.10 64.10 121.95 122.20 126.25
Thermal Conductivity of Upper Reference K A Btu-in./hr ft²*F	714 gm 752 gm	4994 3991 3991 251 195	922 gm	4-98 310 310 230 230
Mean Tempera- ture of Upper Reference	ight: 14.0974 ht: 14.0752	110 111 502 503 1199 1200 1613	ight: 14.1067 ht: 14.0922	97 97 874 877 1328
through Lower Reference AT *F1	Initial Weight: Final Weight:	33.25 33.30 43.74 43.74 93.83 93.58 123.60	Initial Weight: Final Weight:	58.25 58.30 100.48 100.65 103.50
Thermal Conductivity of Lower Reference R ₁ Rtu-in./hr ft²°F	1.1406 in.	517 518 414 415 290 230 228	1.1400 in. 1.1400 in.	541 541 361 361 270 270
Mean Tempera- ture of Lower Reference	Initial Thickness: Final Thickness:	335 9999 13499 13494 13444 13444 13444	Initial Thickness: Final Thickness:	-37 -36 649 650 1100
LT through Specimen	Initial Final Th	12.65 12.65 14.50 14.55 29.57 29.55 36.33	Initial Final Th	21.15 21.25 34.97 34.97 33.35
Thermal Conductivity of Specimen Ks Btu-in./hr ft²on	. 0.750 in.	1395 1395 1303 1304 941 791	= 0.750 in.	1500 1493 1059 1061 854 865
Mean Temperature of Specimen	1 1 2 H 2 S H 2 S H 3 S	454 10854 1088 1467	1 2 1 2	31 31 754 754 1206
Specimen and Time	Specimen: CR-1AB -C-29 Run: 1 Run: 6072-69 Density: 2.452	2:15 2:45 8:30 9:00 12:30 1:00 3:00	Specimen: CR-2AB Run: 1 Run: 6072-73 Density: 2.456	9:50 10:20 2:30 3:10 10:30

Notes: 1.

All measurements made with helium purge. Thermal conductivity $\left(k_S\right)$ of specimen calculated from following equation

$$S = \begin{bmatrix} k_1 \Delta T_1 & + k_2 \Delta T_2 \\ l_1 & l_2 \end{bmatrix} \frac{l_S}{2\Delta T_S}$$

k = thermal conductivity ΔT = temperature drop over gage length l = gage length

and subscripts 1, 2, and s refer to lower reference, upper reference, and specimen, respectively.

Table 24

Thermal Conductivity of Codeposited Pyrolytic Graphite with 25 Percent SiC in the 'C' Direction Measured in Comparative Rod Apparatus with Armco-Iron References

AT through Upper Reference AT?		32.05 32.11 31.33 31.81 76.25		89.90 90.60 54.35 94.52 94.45 91.48
Thermin Conductivity of Upper Reference R ₂ Btu-in./hr ft ² °F	55 gm 46 gm	484 484 380 269 269	89 ஏர	99.0 99.1 123.2 123.2 159.2 173.5
Mean Tempera- ture of Upper Reference	ight: 4.9355 ht: 4.9346	138 138 557 558 1099 1098	ight: 4.9389 ht: 4.9382	179 183 564 565 1153 1153 1432
through Lower Reference	Initial Weight: Final Weight:	28.85 29.35 27.30 27.30 62.83	Initial Weight: Final Weight:	102.40 102.50 59.60 59.56 104.25 104.28 95.75
Thermal Conductivity of Lower Reference R1 Btu-in./hr ft²eF	0.1563 in.	527 527 403 403 313 313	0.1563 in. 0.1563 in.	87.0 87.0 116.0 116.0 145.2 145.2 162.0
Mean Tempera- ture of Lower Reference	Initial Thickness: Final Thickness:	* 4 4 8 8 8 6 9 9 8 1 1 4 4 8 8 8 9 9 9 9 8 9 9 9 9 9 9 9 9 9 9	Initial Thickness: Final Thickness:	2 451 452 925 925 1203
Corrected Tempera- Lore of through Lower Specimen Reference	Initial Thickness: Final Thickness:	16.62 14.19 14.19 34.25	Initial Thickness: Final Thickness:	8.20 8.10 7.29 7.29 21.87 25.84
Thermal Conductivity of Specimen Ks Rtu-in./hr ft²	= 0.750 in. 563 in.	193 195 168 170 122	0.750 in. 0.1563 in.	226 230 195 194 144 127
Mean Temperature of Specimen	$1 = 1 = 0.750$ $1_{S} = 0.1563 \text{ in.}$	67 67 501 502 973	1 = 1 = 0.	94 94 507 508 1039 1315
Specimen and Time	Specimen: CR-1C- C-29 Run: 1 Run: 6072-60 Density: 2.443	2:30 3:00 1:30 7:00 7:30	Specimen: CR-2C- Run: 1 Run: 6072-63 Density: 2.452 gm/cm ³	2:20 2:50 7:45 7:45 12:30 1:00 4:30 5:00

Notes: 1. All measurements made with helium purge.

2. Thermal conductivity (kg) of specimen calcu

Thermal conductivity (k_S) of specimen calculated from following equation

$$k_{S} = \begin{bmatrix} k_{1} \Delta T_{1} & + \frac{k_{2} \Delta T_{2}}{l_{2}} \end{bmatrix} \frac{l_{S}}{2\Delta T_{S}}$$

where

 $k = thermal conductivity \\ \Delta T = temperature drop over gage length \\ l = gage length$

and subscripts 1, 2, and s refer to lower reference, upper reference, and specimen, respectively. *Temperature gradient corrected for grafoil at interfaces from known in house thermal conductivity of grafoil

Table 25

The Thermal Conductivity of Codeposited Pyrolytic Graphite Sleeve with 25 Percent SiC in the 'C' Direction (Measured in Radial Inflow Apparatus Using an Optical Technique)

		1,600				
Run: 1	Outer Surface Tempera- ture PG-SiC °F	Inner Wall Tempera- ture PG-SiC °F		Heat to Calorimeter Btu hr = Outside R	Specimen Mean Temperature °F adius 2.0000 dius 1.8400	
	$.364 \text{ gm/cm}^3$					
12:10	2216	2201	15	208	2208	52.7
1:15	3221	3173	48	618	3197	49.0
2:45	4214	4107	107	1388	4160	49.3
3:30		nated due nner wall		up on calorim	eter blocking	optical
Specimen Sl Run: l	eeve: RI-2	C-C-47				
Run: 1 Run: 5308- Density: 2	115-69-2 .394 gm/cm ³		16	180	2206	58.3
Run: 1 Run: 5308- Density: 2	115-69-2 .394 gm/cm ³	2198	16	180	2206	58.3
Run: 1 Run: 5308- Density: 2 11:00	115-69-2 .394 gm/cm ³ 2214 3108	2198 3055	53	498	2206 3082	48.7
Run: 1 Run: 5308- Density: 2	115-69-2 .394 gm/cm ³	2198				
Run: 1 Run: 5308- Density: 2 11:00	115-69-2 .394 gm/cm ³ 2214 3108 3824 Run termi	2198 3055 3745	53 79	49 8 991	3082	48.7 65.0
Run: 1 Run: 5308- Density: 2 11:00 12:10 2:05 3:00	115-69-2 .394 gm/cm ³ 2214 3108 3824 Run termi	2198 3055 3745 nated due	53 79	49 8 991	3082 3785	48.7 65.0
Run: 1 Run: 5308- Density: 2 11:00 12:10 2:05 3:00	115-69-2 .394 gm/cm ³ 2214 3108 3824 Run termi view of i	2198 3055 3745 nated due	53 79	49 8 991	3082 3785	48.7 65.0
Run: 1 Run: 5308- Density: 2 11:00 12:10 2:05 3:00 Run: 2 RI- Run: 5308-	115-69-2 .394 gm/cm ³ 2214 3108 3824 Run termi view of i 2C-C-47 117-A24	2198 3055 3745 nated due nner wall	53 79 to build-	498 991 up on calorim	3082 3785 eter blocking	48.7 65.0 optical
Run: 1 Run: 5308- Density: 2 11:00 12:10 2:05 3:00 Run: 2 RI- Run: 5308- 9:45	115-69-2 .394 gm/cm ³ 2214 3108 3824 Run termi view of i 2C-C-47 117-A24	2198 3055 3745 nated due nner wall	53 79 to build-	498 991 up on calorim	3082 3785 eter blocking 2553	48.7 65.0 optical

The thermal conductivity was calculated from the Equation:

$$K = \frac{\ln (R_2/R_1)}{2\pi L} \frac{Q}{\Delta T}$$

where

K = thermal conductivity

Q = heat to calorimeter

ΔT = temperature difference between outside and inside radif
L = gage length of calorimeter 0.500 in.

Notes:

All measurements made in 1 atm helium purge after evacuating and back filling furnace twice with helium

Table 26

Electrical Resistivity of Codeposited Pyrolytic Graphite with 25 Percent and 15 Percent Silicon Carbide in the 'a-b' Direction

Time	Specimen Temperature °F	Current Through Circuit Amps	Closed Circuit minus Open Circuit Potential mv	Electrical Resistivity µohm-cm
	Specimen: ER Run: 1 Run		s sic)	
8:30	76	0.3 -0.3	5.55 -5.47 Avg. 5.51	1557
9:15	195	0.3 -0.3	5.12 -5.15 Avg. 5.14	1453
9:35	393	0.3 -0.3	4.38 -4.37 Avg. 4.37	1237
10:00	593	0.3 -0.3	3.89 -3.75 Avg. 3.82	1080
10:30	846	0.3 -0.3	3.24 3.31 Avg. 3.28	927
11:25	1173	0.3 -0.3	2.76 2.81 Avg. 2.79	789
12:10	1458	0.3 -0.3	2.55 2.56 Avg. 2.56	724
12:35	1638	0.3 -0.3	2.37 2.40 Avg. 2.38	674
1:00	1810	0.3	2.32 2.33 Avg. 2.32	657
2:20	Cool Down Poi	0.3 -0.3	5.55 5.44 35.50	1555

Table 26 (Continued)

	·			*
Time	Specimen Temperature °F	Current Through Circuit Amps	Closed Circuit minus Open Circuit Potential mv	Electrical Resistivity µohm-cm
	Specimen: ER	-1AB-C31 (2	5% SiC)	
9:30	73	0.3	$ \begin{array}{r} 21.63 \\ -21.63 \\ \hline \text{Avg.} & 21.63 \end{array} $	6114
10:00	190	0.3	20.12 -19.16 Avg. 19.64	5552
10:20	359	0.3	16.89 -16.49 Avg. 16.69	4718
10:40	552	0.3 -0.3	13.65 -13.47 Avg. 13.56	3833
11:00	794	0.3 -0.3	10.84 -10.86 Avg. 10.85	3067
11:20	1021	0.3	8.98 - 8.88 Avg. 8.93	2524
11:40	1283	0.3 -0.3	7.45 - 7.43 Avg. 7.44	2103
12:00	1590	0.3	6.34 - 6.52 Avg. 6.43	1818
12:25	1799	0.3	5.98 - 5.05 Avg. 6.02	1702
	Cool Down F	Point (RT)		
1:10	78	0.3	21.60 21.16 Avg. 21.38	6043

Note:

Negative sign indicates that the direction of the current through the circuit was reversed.

APPENDICES

Appendix A	Thermal Expansion to 1800°F
Appendix B	Thermal Expansion to 5500°F
Appendix C	A Comparative Rod Apparatus for Measuring Thermal Conductivity to 2000°F
Appendix D	Thermal Conductivity to 5500°F by Radial Inflow Method

APPENDIX A

THERMAL EXPANSION TO 1800°F

Thermal expansion measurements are made utilizing quartz tube silatometers of the Bureau of Standards design. The dial gages (i. c. Ames Co., Model 212, Shockless) are graduated in 0.0001-inchesivisions with a total range of 0.500 inch. The manufacturer's stated mechanical accuracy for any given reading is ± 0.0001 inch at any point in the range. This accuracy has been checked with a precision micrometer.

The extensions from the dial, which are made of stainless steel, are finned to facilitate cooling. The temperatures of the fins are continually monitored and air cooled when necessary to eliminate differential expansion between the dial gage mount and the extension; see Figure 1.

For temperatures above room temperature, each dilatometer is meated by an individual heater. The temperature of the heater is maintained by a manual setting of a variable voltage transformer.

Cold specimen temperatures are obtained by use of a Dewar flask filled with dry ice and trichloroethylene. The flask surrounds the dilatometer tubes and the cold liquid level rises to a height above the specimens.

Liquid nitrogen is used in the Dewar flask for temperatures down to -300°F. A cooling coil has also been designed to provide better control of temperatures in the cryogenic range.

Thermocouples are placed at each end and the center of the specimens to monitor the temperature throughout. The specimens are nominally 1/2-inch diameter by 3 inches in length with the ends rounded on a 3-inch radius. Other diameters and cross-sectional configurations are employed where necessary due to configuration of supplied material.

To calibrate the dilatometers we employ a fused silica specimen, the expansion of which is known and is shown in Figure 2.

The accuracy of the apparatus to 1800°F has been checked by running the NBS copper standard and other in-house standards of graphite and nickel. Figures 3, 4, and 5 include the expansion values measured in the quartz dilatometers on these standards. Note the excellent agreement between the measured values and the data reported by NBS on the copper. For the graphite and nickel standards, good agreement was observed between the values measured in the quartz dilatometer and that reported in the literature and the values measured at SRI by optically tracking.

The above data and our experience have demonstrated the excellent precision and accuracy of this equipment. We have observed no systematic uncertainty with the equipment and, based on 5 runs with the graphite standard, the random uncertainty is $\pm 0.05 \times 10^{-3}$ in./in. at 1500°F with a 95 percent confidence interval.

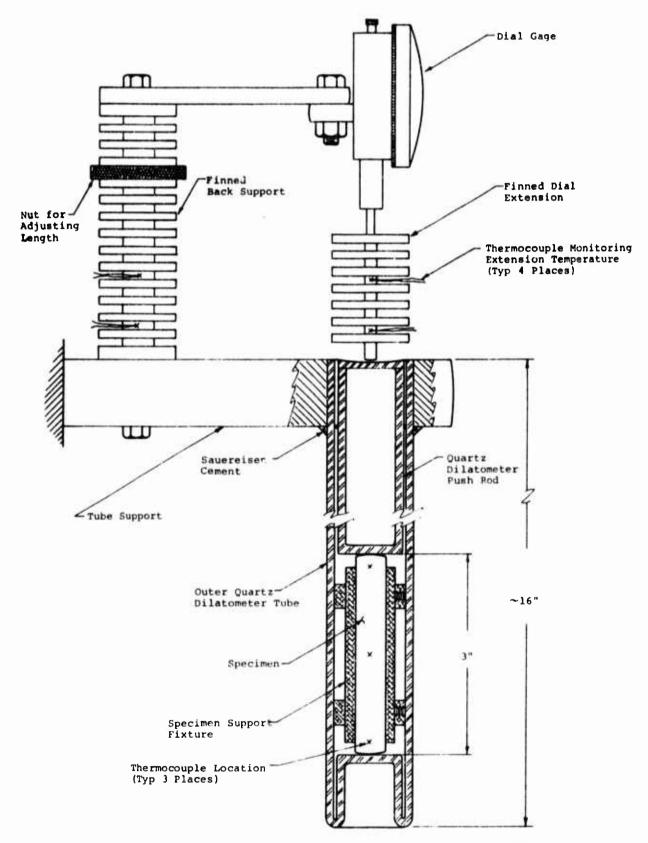
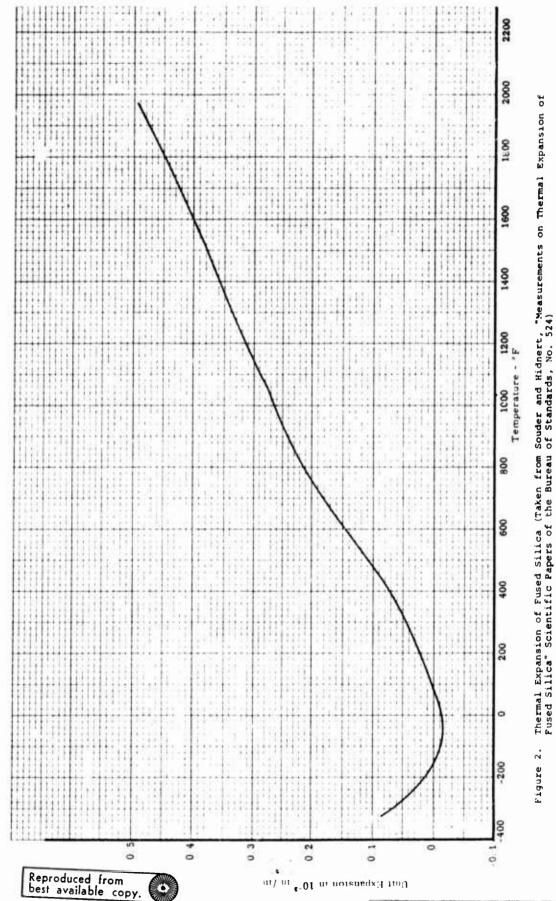
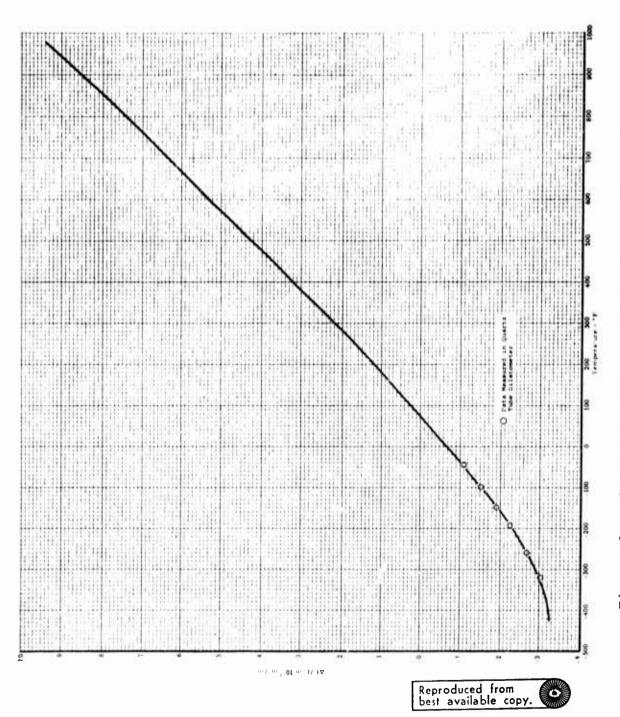


Figure 1. Assembly of Quartz Tube Dilatometer for Thermal Expansion Measurements



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Thermal Expansion of NBS Copper Standard SRM 736 . س

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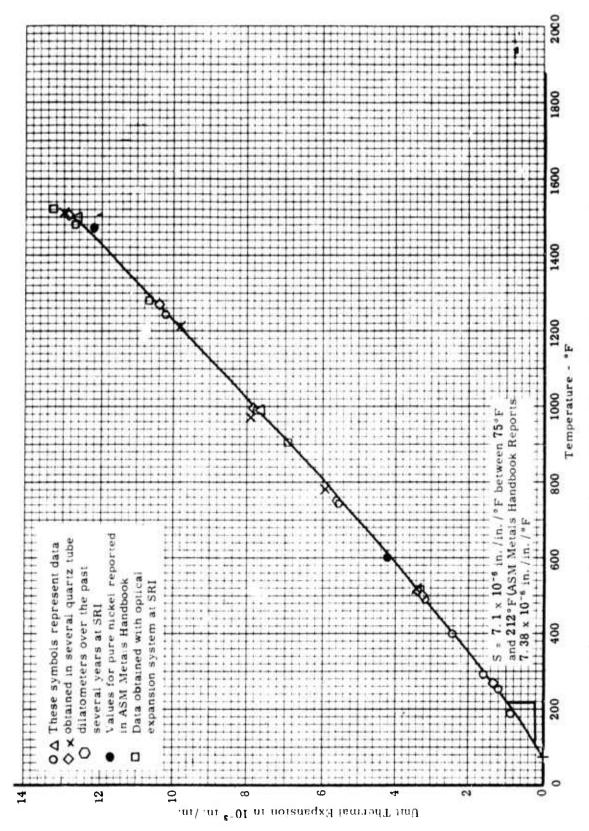


Figure 4. Expansion of "A" Nickel (Calibration Standard Specimen)

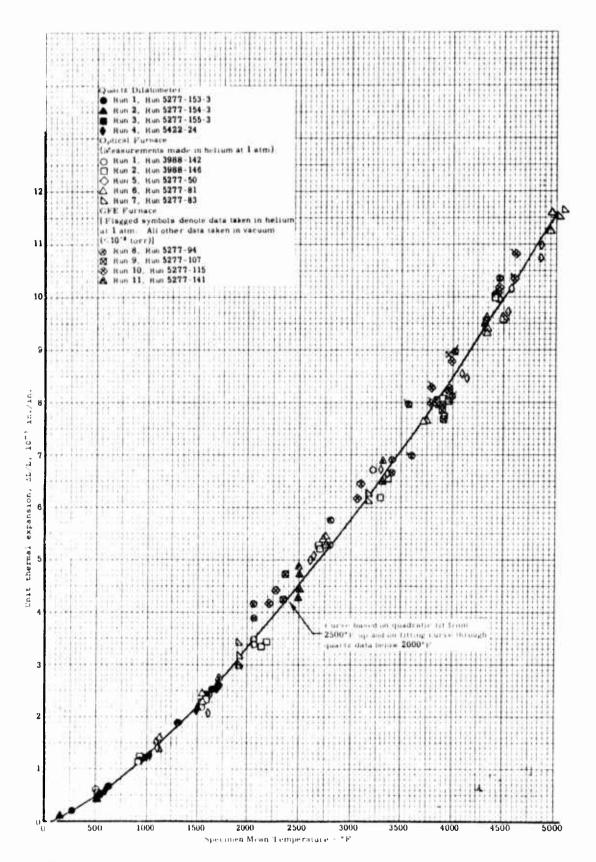


Figure 5. Thermal Expansion of ATJ Graphite (wg) Standard No. 5 by In-House Optical Calibration

APPENDIX B

THERMAL EXPANSION TO 5500°F

Thermal expansion is measured in a graphite tube dilatometer developed by Southern Research Institute for performance to 5500°F, see Figure 1. The specimen required is about 1/2" diameter and 3" long, although the exact size can vary somewhat if it appears desirable from the standpoint of specimen availability. Specimens 3/4" in diameter and only 1/4" thick can be evaluated, but with a reduced precision. Discs can be stacked to provide more length in many cases. Of course, specimens can always be pinned together from smaller pieces to provide both length and columnar strength.

In the dilatometer, the specimen rests on the bottom of the cylinder with a graphite extension rod resting on the specimen to extend to the top of the cylinder. When required, tungsten pads are inserted at the ends of the specimens to eliminate graphite diffusion from the dilatometer parts into the specimen. This entire assembly is inserted into one of the 5000°F furnaces described in another brochure.

The motion of the specimen is measured by a dial gage attached to the upper end of the cylinder with the stalus bearing on the extension rod. The system accurately indicates total motions of 0.0001" - or less than 0.00004" per inch of specimen.

Either a helium or an argon environment can be employed. Nitrogen has been used on occasion. The equipment will permit operation at hard vacuums, but this procedure is rarely used.

A CS graphite, which has a fairly low expansion relative to other grades of graphite, is used as the material for the dilatometer. Prior to calibrations, the dilatometers are heat soaked to a temperature several hundred degrees above the maximum temperature to which they would be exposed during normal service. Dimensional stability is confirmed by measuring the lengths of the dilatometer tube and rod after each run. Past experience has shown that following the initial heat soak the expansion is reproducible in subsequent repeated cycles to lower temperatures. Reproducibility is also confirmed by repeated runs on standards.

To calibrate the dilatometers we have developed in-house primary and secondary standards of ATJ graphite. ATJ graphite was selected as a standard because of our vast experience with it, its stability after repeated exposure to high temperatures, and its relatively low expansion.

The true expansion of the primary standard was determined by a direct optical technique using a traveling Gaertner telescope. total error in the telescope readings, based on calibration data, was estimated to be 0.2×10^{-3} in./in. For the direct optical measurements, the 3.5 inch long specimen was heated in a graphite furnace, and the expansion was determined by sighting on "knife" edges machined on the ends of the specimen. Typically a total of ll runs have been made in two different furnaces both in vacuum and helium environments. The two environments are used to check effects of refraction as reported in the literature. standard was then machined to the configuration of a regular dilatometer specimen and several runs were made in our precision quartz dilatometers. The optical expansion data were fitted to a quadratic equation over the temperature range from 2500°F to 5000°F using the method of least squares and statistically analyzed to determine the uncertainty (primarily the scatter). Below 2500°F, the quartz dilatometer data were fitted by hand since the uncertainty of this apparatus has been well established, and the imprecision is small ($<0.1 \times 10^{-3}$ in./in.). A typical plot of all data points with the curve fit is shown in Figure 2.

A check of the expansion of the standard was obtained by making runs on round robin specimens of various graphites and synthetic sapphire which had been previously evaluated by others, including the National Bureau of Standards. Our data on these specimens agreed within a 2.5 percent random difference with the data reported by the other laboratories.

After establishing the expansion of the ATJ standard, several graphite dilatometers were then calibrated by making runs on this standard. These dilatometers were used to establish the expansion of secondary standards (also ATJ graphite) which are used to calibrate new dilatometers and to make periodic checks on dilatometers currently in service. This use of secondary standards thus minimizes the wear and tear on the primary standard and prolongs its life.

Table 1 lists the uncertainties in the dilatometer measurements in 10^{-3} in./in. Observe that most of the uncertainty is in the expansion of the standard and includes both random and systematic uncertainties. Other sources of uncertainty, resulting from such factors as dial gage and temperature measurement, are small amounting to less than 0.2×10^{-3} in./in. at any temperature. The precision in the dilatometer measurements is quite good and amounts to about 0.1×10^{-3} in./in. From Table 1, it can be seen that the maximum total uncertainty, which occurs at a temperature of 4500° F, is $\pm 0.45 \times 10^{-3}$ in./in. For a low expansion graphite, such as ATJ, this amounts to an uncertainty of ± 4.5 percent at 4500° F (see Figure 2). For graphites having higher expansions, the percentage uncertainty would be lower.

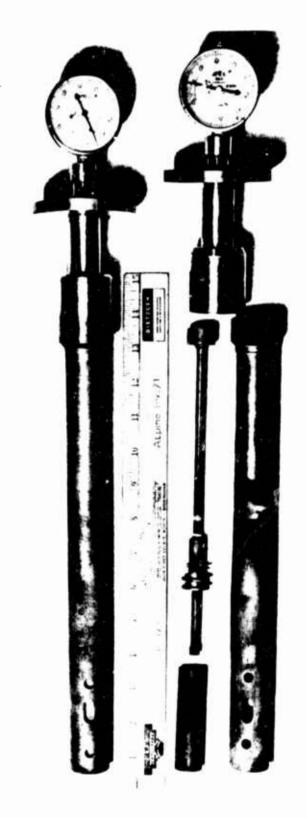


Figure 1. Picture of the Graphite Dilatometer Tubes for Measuring Thermal Expansion to 5500°F

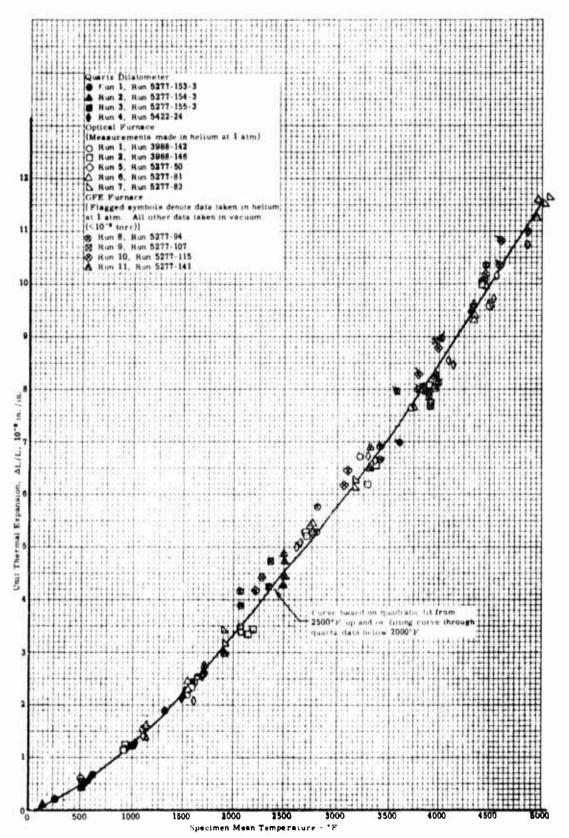


Figure 2. The Thermal Expansion of ATJ Graphite (wg) Standard No. 5 by In-House Optical Calibration

Table 1
Uncertainty in Thermal Expansion Measurements
Made in Graphite Dilatometers

	Uncertai Expansio Standa in 10 ⁻¹	on of	Random Uncertainty	Total Uncertainty in Dilatometer Measurements				
Temperature °F	Random Uncertainty (See Note 1)	Systematic Uncertainty (See Note 2)	in Dilatometer Measurements in 10 ⁻³ in./in.	from all in 10 ⁻³	Sources in./in.			
500	±0.04	0	±0.03	0.05	0.05			
1000	±0.04	0	±0.03	0.05	0.05			
1500	±0.05	0	±0.04	0.07	0.07			
2000	±0.17	+0.03	±0.05	0.21	0.18			
2500	±0.11	+0.20	±0.07	0.33	0.13			
3000	±0.12	+0.21	±0.08	0.35	0.14			
3500	±0.19	-0.02	±0.10	0.21	0.23			
4000	±0.14	-0.09	±0.12	0.18	0.27			
4500	±0.14	±0.25	±0.14	0.45	0.45			
5000	±0.19	-0.12	±0.16	0.25	0.37			

Notes: 1. 95% confidence limits.

2. Represents deviation between average measured value and least squares curve through all data.

APPENDIX C

A COMPARATIVE ROD APPARATUS FOR MEASURING THERMAL CONDUCTIVITY TO 2000°F

Southern Research Institute's comparative rod apparatus is used to measure thermal conductivities of a wide variety of materials from -300°F to 2000°F. This apparatus, shown schematically in Figure 1, consists basically of two cylindrical reference pieces of known thermal conductivity stacked in series with the cylindrical specimen. Heat is introduced to one end of the rod, composed of the references and specimen, by a small electrical heater. A cold sink or heater is employed at the opposite end of the rod as required to maintain the temperature drop through the specimen at the preferred level. Cylinders of zirconia may be inserted in the rod assembly to assist in controlling the temperature drop. Radial losses are minimized by means of radial guard heaters surrounding the rod and consisting of three separate coils of 16, 18 or 20-gage Kanthal wire wound on a 2 or 4-inch diameter alumina core. The annulus between the rod and the guard heaters is filled with diatomaceous earth, thermatomic carbon, bubbled alumina or zirconia powder. Surrounding the guard is an annulus of diatomaceous earth enclosed in an aluminum or transite shell.

The specimens and references (see Figure 2) are normally 1-inch diameter by 1-inch long. Thermocouples located 3/4 inch apart in radially drilled holes measure the axial temperature gradients. Thermocouples located at matching points in each guard heater are used to monitor guard temperatures, which are adjusted to match those at corresponding locations in the test section.

In operation, the apparatus is turned on and allowed to reach steady state. The guard and rod heaters are adjusted to minimize radial temperature gradients between the rod and guard sections consistent with maintaining equal heat flows in the references. Temperatures are measured on a Leeds and Northrup Type K-3 potentiometer, and the temperature gradients calculated. A typical temperature profile in the test section is shown in Figure 3.

The thermal conductivity of the specimen is calculated from the relation

$$K_{s} = \frac{K_{1} \Delta T + K_{2} \Delta T}{2\Delta T_{s}} \frac{\Delta X_{s}}{\Delta X_{r}}$$

where K_1 and K_2 are the thermal conductivities of the upper and lower references; ΔT_1 , ΔT_2 and ΔT_8 are the temperature differences in the upper and lower references and specimen, respectively; ΔX_8 and ΔX_7 are the distances between thermocouples in the specimen and references.

Note that for purely axial heat flow, the products $K_1\Delta T_1$ and $K_2\Delta T_2$ should be equal. Due to imperfectly matched guarding and other factors, this condition is seldom attained in practice; therefore, the average of the two values is used in the calculations. Their difference is maintained as small as possible, usually within 5 percent of the smaller.

For identical specimens, the radio $\Delta X_s/\Delta X_r$ should be unity but may vary due to the uncertainty in hold locations. To prevent introducing an additional error in calculations, ΔX is determined as follows: the depth of the hole is measured by inserting a snugly fitting drill rod in the hole, measuring the projecting length and subtracting it from the total length of the rod. The slope, or angle the hole makes with the perpendicular to the specimen axis, is determined by making measurements to the face of the hole and the outer end of the drill rod with respect to a datum plane, using a dial gage. From these measurements, the location of the bottom of the hole can be calculated.

Generally, measurements with the comparative rod apparatus are performed in an inert helium environment. The apparatus can also be operated in vacuum and at gas pressures of up to 100 psig. We have had experience operating under all conditions.

The primary reference materials which we use are Code 9606 Pyroceram and Armco iron for measurements on materials with low and high thermal conductivities, respectively. Primary standard reference sets are kept and are used to calibrate other references made from the same materials. The standards of Code 9606 Pyroceram were made from a batch of material which NBS purchased shortly after their measurements on a sample of Code 9606 Pyroceram. The curve which Flynn presented for the thermal conductivity of the Pyroceram is given in Figure 4. Note that the curve is in good

Robinson, H. E. and Flynn, D. R., Proceedings of Third Conference on Thermal Conductivity, pages 308-321, 1963 (with author's permission)

agreement with the recommended values from NSRDS-NBS 8^2 . The standards of Armco iron were made from the stock which was used in the round-robin investigations from which Powell³ developed the most probable values for Armco iron. The curve used for the Armco iron standards is shown in Figure 5. Powell estimated the uncertainty to be within ± 2 percent over the temperature range from 0° to 1000° C. Note in Figure 5 that numerous evaluations of Armco iron from other batches of material have agreed within ± 3 percent (coefficient of variation about curve) with Powell's original data.

In addition to Code 9606 Pyroceram and Armco iron, several other materials have been used as references. These include copper for high conductivity specimens, 316 stainless steel for specimens of intermediate thermal conductivity and Teflon or Pyrex for low conductivity materials.

Copper references have been calibrated against Armco iron and excellent agreement with literature data has been obtained. Thermal conductivity values obtained from calibrations of 316 stainless steel against Pyroceram, Armco iron and a set of 316 stainless steel standards are presented in Figure 6. Note the consistency of the data obtained with the three different sets of references. The coefficient of variation of the data shown in Figure 6, about the curve value, was ±3.3 percent. These data indicate the internal consistency of the stainless steel and the reference materials. Note that the thermal conductivity values for 316 stainless steel presented in Figure 6 lie between values reported by several steel manufacturers and Lucks and Deem.

The calibrations indicate that for materials with moderate to high thermal conductivities the apparatus operates with a precision of about ±3 percent and a total uncertainty of about ±5 percent at temperatures above 0°F if temperatures between the guard and test section are closely matched. Below 0°F, the precision achieved to date has been about ±7 percent with a total uncertainty of about ±10 percent. We anticipate that the precision and uncertainty at cryogenic temperatures can be improved by additional calibrations.

Powell, R. W., C. Y. Ho and P. E. Liley, <u>Thermal Conductivity</u> of <u>Selected Materials</u>, NSRDS-NBS 8, Department of Commerce, <u>November 25, 1966.</u>

Powell, R. W., Proceedings of Third Conference on Thermal Conductivity, pages 322-341, 1963.

[&]quot;WADC TR58-476, "The Thermophysical Properties of Solid Materials", Armour Research Foundation, November, 1960.

Some additional data obtained on the comparative rod apparatus are shown in Figures 7 and 8. Figure 7 shows thermal conductivity data for ATJ graphite, with grain, using Armco iron as the referene material. These data show excellent agreement with earlier data obtained here and by other sources 5-7. The maximum scatter of the comparative rod points was about 5 percent.

Figure 8 shows data for thermocouple grade constantan obtained on the comparative rod apparatus using Armco iron references and on Southern Research Institute's high temperature radial inflow apparatus. Note the excellent agreement. These data also show close agreement with data obtained by Silverman' on an alloy of very similar composition.

⁵ ASD-TDR-62-765, "The Thermal Properties of Twenty-Six Solid Materials to 5000°F or Their Destruction Temperatures," Southern Research Institute, August, 1962.

⁶ Pears, C. D., Proceedings of Third Conference on Thermal Conductivity, 453-479, 1963.

NSRDS-NBS 16, "Thermal Conductivity of Selected Materials", Part 2, by C. Y. Ho. R. W. Powell and P. E. Liley, National Bureau of Standards, 1968.

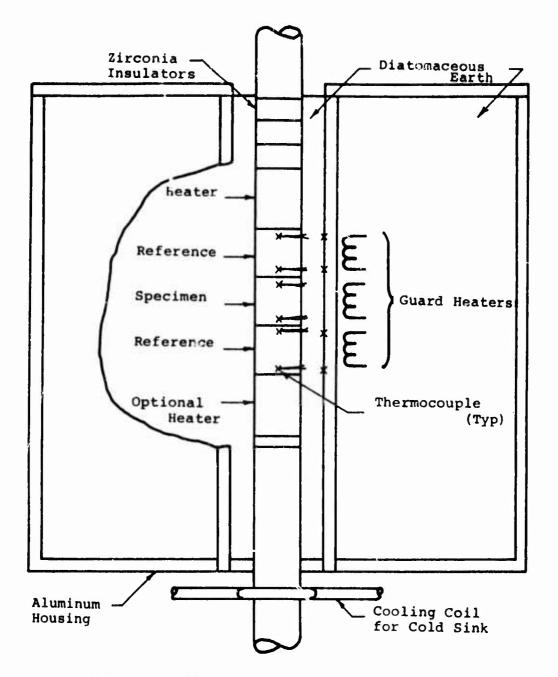
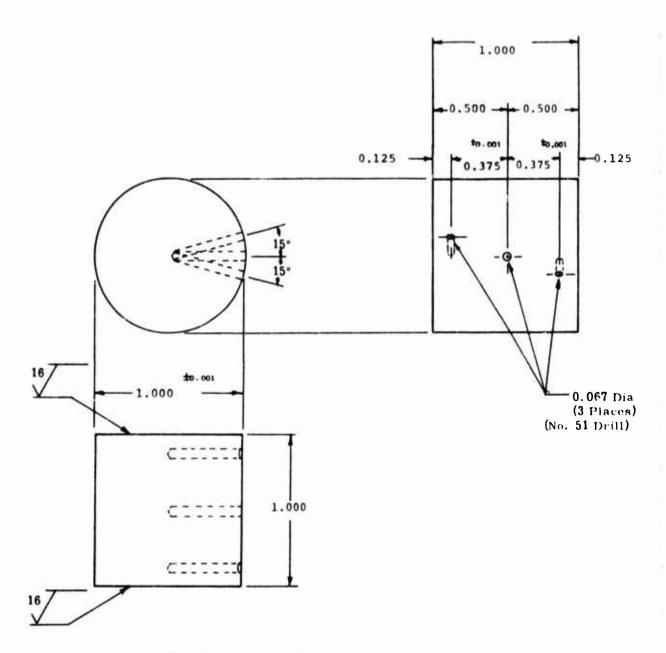


Figure 1. Schematic of Comparative Rod Thermal Conductivity Apparatus



Note: All dimensions ± 0.005 except where noted

Figure 2. Drawing of Specimen for Thermal Conductivity Measurements in Comparative Rod Apparatus to 1800°F

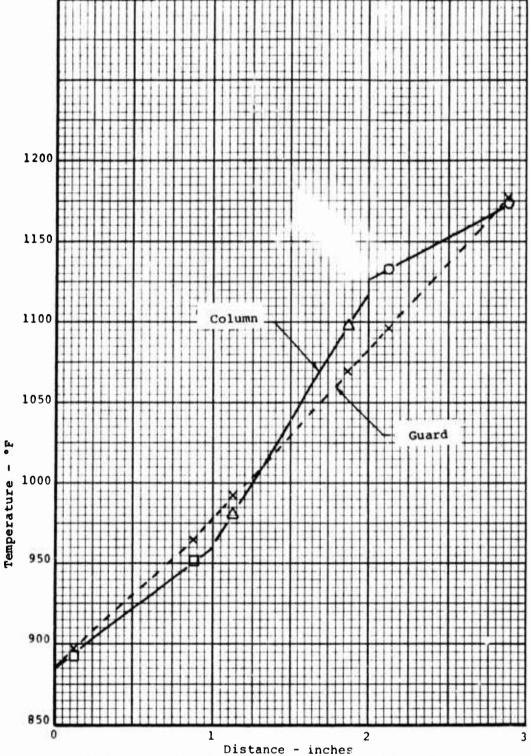
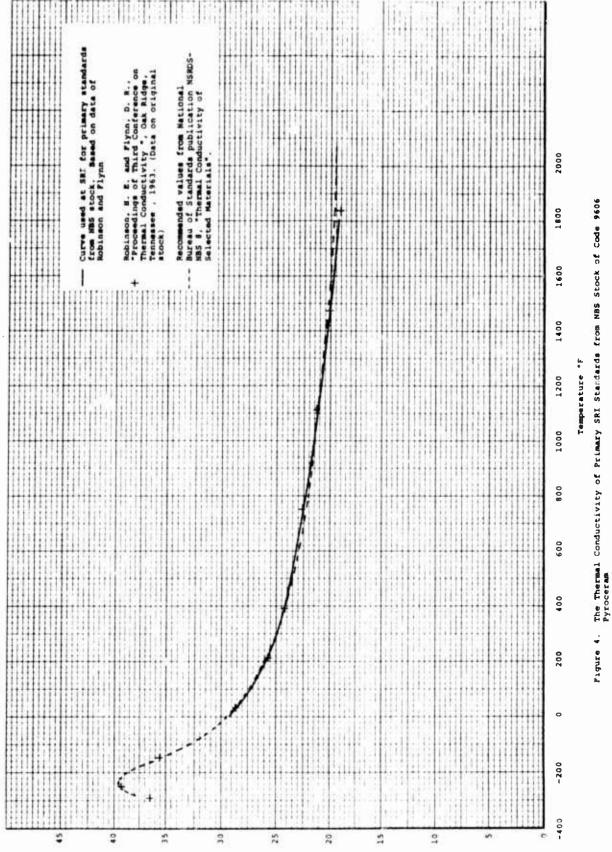
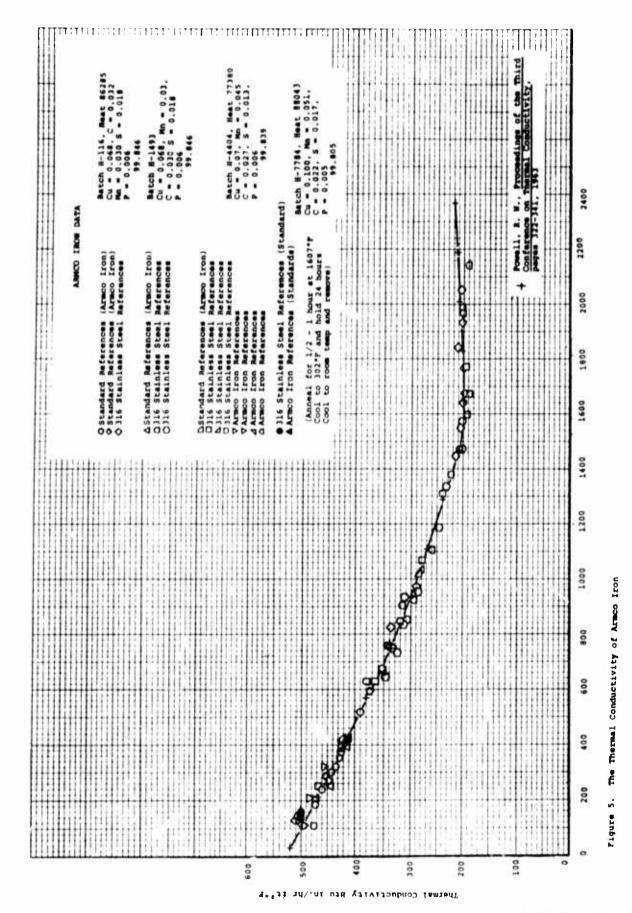


Figure 3. Typical Temperature Profile in Test Section



Thermal Conductivity - Btu in./hr ft 10F

Figure 4.



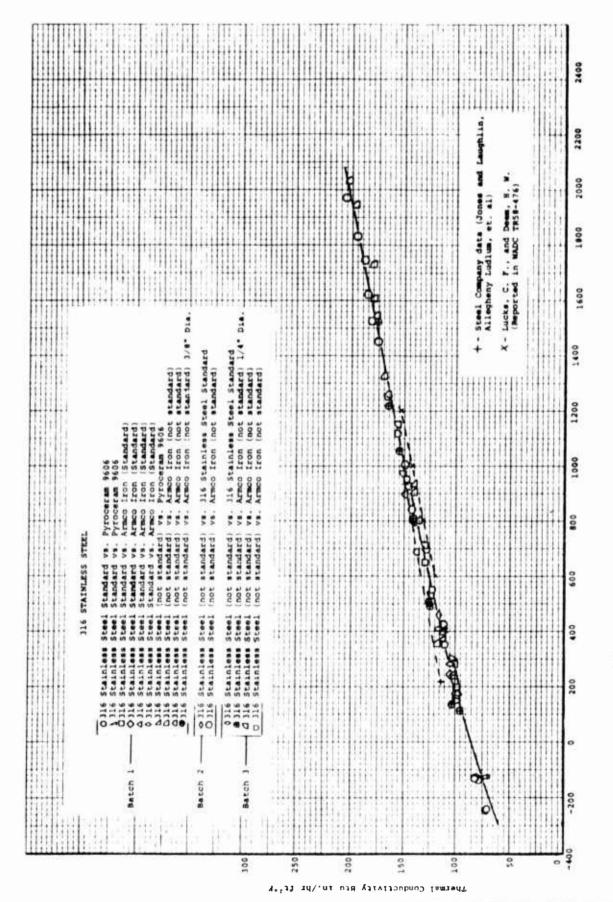
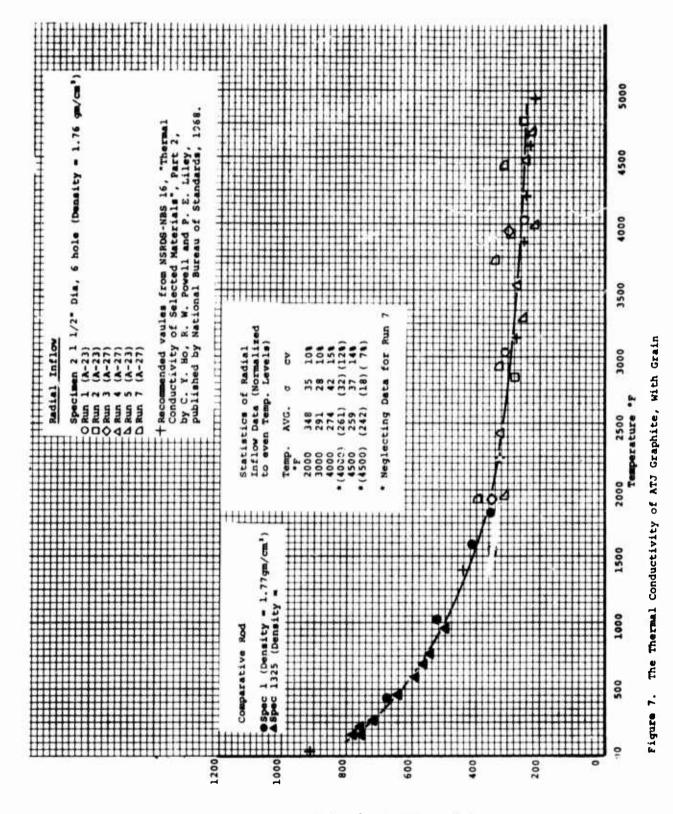


Figure 6. The Thermal Conductivity of 316 Stainless Steel



Thermal Conductivity - Btu in./hr ft**F

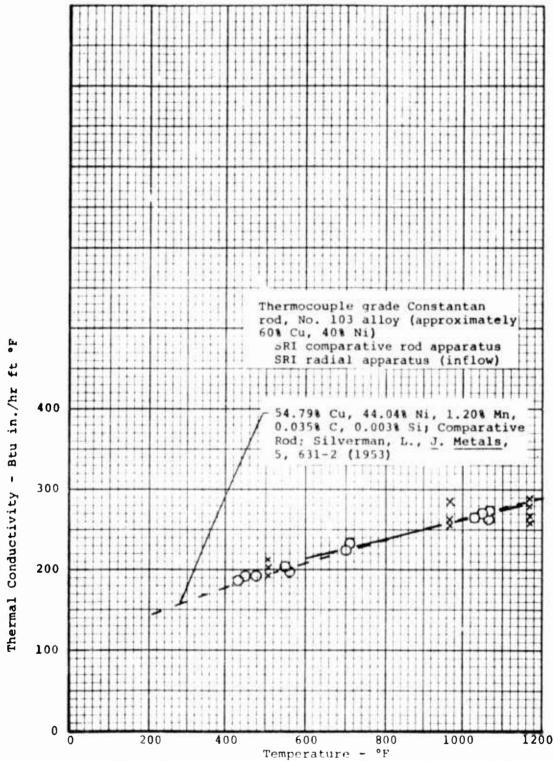


Figure 8. The Thermal Conductivity of Thermocouple Grade Constantan Rod

THERMAL CONDUCTIVITY TO 5500°F BY RADIAL INFLOW METHOD

The thermal conductivity is determined with a radial heat inflow apparatus that utilizes a central specimen 1" long. apparatus is normally employed for measurements over the temperature range from 1500°F to 5500°F. Comparative rod apparatus is used at temperatures below 1500°F where radiant heating is less The radial inflow apparatus gives a direct measurement of the thermal conductivity rather than a measurement relative to some standard reference material. A picture of the apparatus ready to be installed in the furnace is shown in Figure 1. The furnace and associated equipment for the thermal conductivity work is shown in Figure 2. In addition to the specimen, the apparatus consists primarily of (1) a water calorimeter that passes axially through the center of the specimen, (2) guards made from the same specimen material at both ends of the specimens to reduce axial heat losses, (3) sight tubes that allow the temperature at selected points in the specimen to be determined either by thermocouples or optical pyrometer and (4) an external radiant heat source (see Figure 3). The water calorimeter provides a heat sink at the center of the specimen to create a substantial heat flow through the specimen and allows the absolute value of the heat flow to be determined. couples mounted 1/2" apart in the calorimeter water stream measure the temperature rise of the water as it passes through the gage portion of the specimen. By metering the water flow through the calorimeter, it is possible to calculate the total radial heat flow through the 1/2" gage section of the specimen from the standard relationship $\Omega = MC\Delta T$. M is the weight of water flowing per hour, C is the specific heat of water and ΔT is the temperature rise of the water as it passes through the gage section.

The standard specimen configuration is shown in Figure 4. The specimen is 1.062" O.D. x 0.250" I.D. x 1" long. Holes 0.073" in diameter are drilled on radii of 0.233 and 0.437" to permit measurement of the radial temperature gradient. In specimens which are anisotropic in the diametral plane (for example, certain graphites) a second pair of holes is drilled 90° to the first pair. The diameters joining each pair of holes is located to coincide with the principal planes of anisotropy in the material.

A 1/2" long upper guard and a 1/2" long lower guard of specimen material are placed above and below the 1" long specimen to maintain a constant radial temperature gradient throughout the entire specimen length and thereby prevent axial heat flow in the

specimen. The outer ends of the specimen guards are insulated with graphite tubes filled with thermatomic carbon. These tubes also hold the specimen in alignment. The combined effect of specimen guards and thermatomic carbon insulation permits a minimum axial temperature gradient within the specimen. This gradient is not detectable by optical pyrometer readings. Visual inspection of the specimens after runs have verified that no large axial temperature gradient exists in the specimen. The guards, made of specimen material, display axial distortion of the isothermal lines for approximately 1/4" from the outer ends before reaching an apparent constant axial temperature.

When sufficient material is available the alternate specimen configuration shown in Figure 5 is employed. This specimen, being 1.5" in diameter, provides a larger gage length (0.357") between temperature wells and allows the installation of three holes on each radius without excessively distorting the radial temperature profiles. Thus this specimen configuration permits a more precise measurement of the average temperature at each radial location. As with the smaller specimen, the location of the temperature wells must be altered for transversely anisotropic specimens.

The annulus between the specimen inside diameter and the 7/32" outside diameter of the calorimeter tube is packed with either copper granules, graphite or zirconia powder. This packing provides a positive method for centering the calorimeter within the specimen and promotes good heat transfer between specimen and calorimeter.

Temperatures up to 2000°F are measured with Chromel/Alumel thermocouples inserted into the specimen through the sight tubes. At high temperatures, the temperatures are measured through the certical sight tubes using a right-angle mirror device and optical pyrometer.

In Figures 1 and 3 showing a typical conductivity calorimeter apparatus ready for insertion into a furnace for a run, a water-cooled copper section can be seen at the top of the unit. This section provides permanent sight tubes to within about 2-1/2" of the guard specimen, in addition to a permanent mount for the right-angle mirror device used with the optical pyrometer. Within the short zone between the water-cooled section and the top guard, thin-walled graphite sight tubes are fitted. The remainder of the annulus is filled with thermatomic carbon insulation.

During thermal conductivity runs, the following data are recorded: (1) power input, (2) specimen face temperature, (3) specimen temperatures in the gage section at the two radii, (4) temperature of the calorimeter water at two points 1/2" apart axially within the specimen center and (5) water flow rate through the calorimeter. At least 5 readings are made at each general temperature range to determine the normal data scatter and to minimize the error that might be encountered in a single reading.

All thermocouple readings are measured on a Leeds and Northrup K-3 null balance potentiometer used in conjunction with a galvanometer of 0.43 microvolts per mm deflection sensitivity. All optically measured temperatures are read with a Leeds and Northrup Type 8622 optical pyrometer. The flow rate of the calorimeter water is measured with a Fischer and Porter Stabl-Vis Flowrater.

The thermal conductivity values are computed from the relation

$$K = \frac{Q \ln \frac{r_{2}}{r_{1}}}{2\pi L (T_{r_{2}}^{-}T_{r_{1}}^{-})}$$

where

Q = the heat flow to and measured by the calorimeter

 r_2 = the radius to the outer temperature well $r_1 =$ the radius to the inner temperature well

 T_r , = temperature at r,

 $T_{r_1}^{r_2}$ = temperature at $r_1^{r_2}$ = the gage length over which the calorimeter ΔT is measured, for our present calorimeter is 1/2 inch

Based on an extensive error analysis and calibrations on homogeneous isotropic materials of known thermal conductivities, such as Armco iron and tungsten, the precision (coefficient of variation) in the measurements has been established at ±7 percent over the temperature range. For multiple runs on samples having similar properties, the uncertainty in a smooth curve through the data can be established to within ±7 percent. A detailed error analysis has been presented in a paper by Mann and Pears. 1

Data obtained here on several high temperature materials are presented in Figures 6, 7 & 8. Figure 6 is a plot of data obtained here on tungsten. The specimen for these determinations were fabricated from stacks of 0.060 inch washers cut from hot rolled sheet stock. Also plotted are values reported by other investigators including "recommended values" given by Powell, Ho and Liley based on a compilation of 103 sets of data. Agree-

¹ Mann, W. H. Jr., and C. D. Pears, "A Radial Heat Flow Method for the Measurement of Thermal Conductivity to 5200°F", presented at the Conference on Thermal Conductivity Methods, Battelle Memorial Institute, October 26-28, 1961.

Powell, R. W., C. Y. Ho and P. E. Liley, "Thermal Conductivity of Selected Materials", NSRDS-NBS 8, National Standard Reference Data Series - National Bureau of Standards - 8, 1966, pp. 11, 54-59.

the recommended values is excellent throughout the temperature range.

Figure 7 shows data obtained here on ATJ graphite, with grain. his material is premium grade, medium grain graphite having a density range of 1.73 to 1.78 gm/cm³. The crosses (+) shown in the figure are "recommended values" given by Ho, Powell and Liley. Again agreement is excellent.

Figure 8 shows data obtained on AXM-5Ql. These data were obtained under a program sponsored by the Air Force Materials Laboratory to develop high temperature thermal conductivity standards. Measurements were made on this material by four laboratories in addition to Southern Research Institute. The bands shown in Figure 8 represent the range of data reported by the other participating organizations. A complete presentation and discussion of the data are given in AFML-TR-69-2.

³Ho, C. Y., R. W. Powell and P. E. Liley, "Thermal Conductivity of Selected Materials, Part 2," NSRDS-NBS 16 National Standard Reference Data Series - National Bureau of Standards-16, pp. 89-128.

[&]quot;AFML-TR-69-2, "Development of High Temperature Thermal Conductivity Standards" submitted by Arthur D. Little, Inc., under Contract AF33(615)-2874, 1969, pp. 115-127.

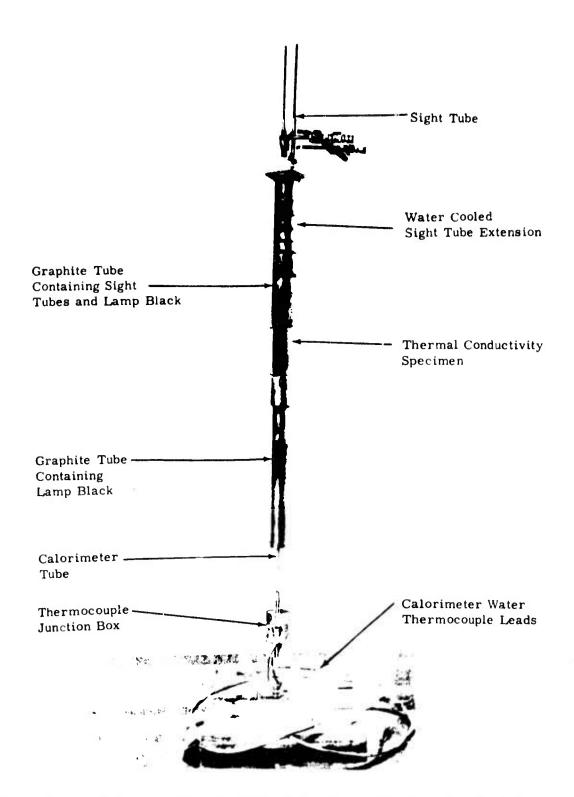
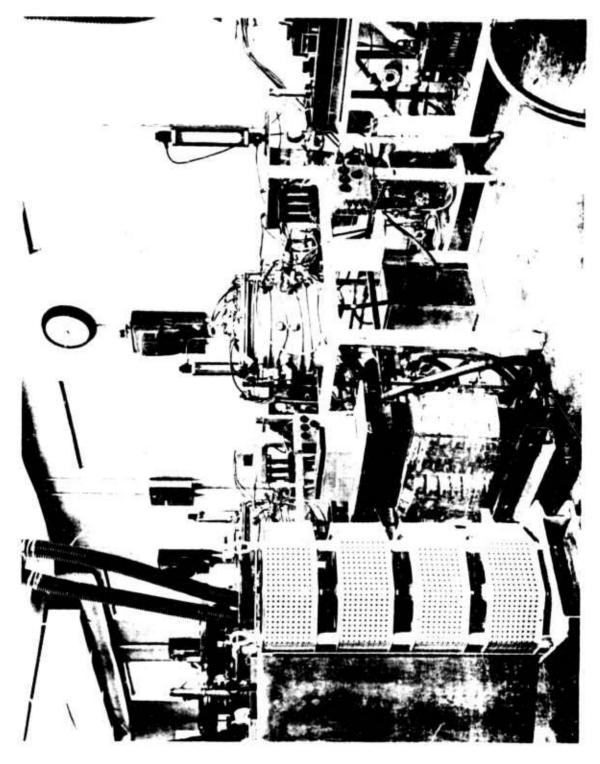


Figure 1. Picture of the Radial Thermal Conductivity Apparatus



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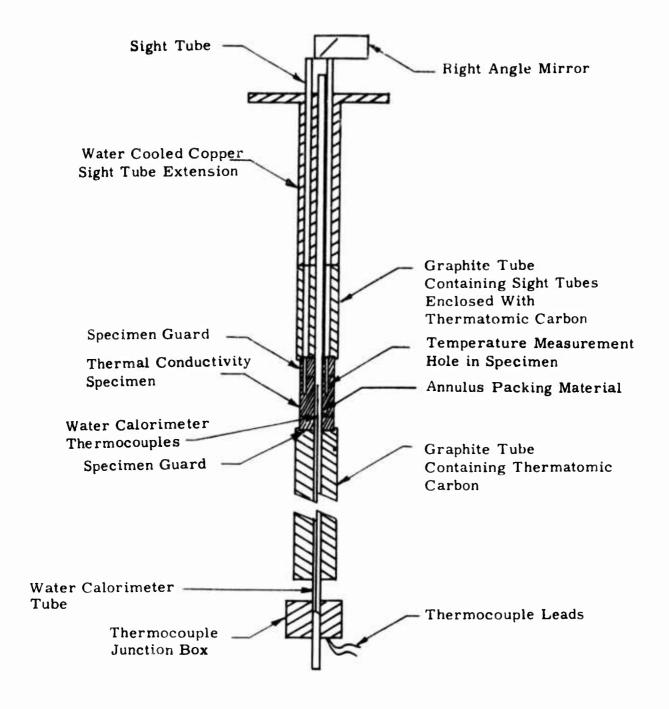


Figure 3. Cross-section Schematic of the Thermal Conductivity Apparatus

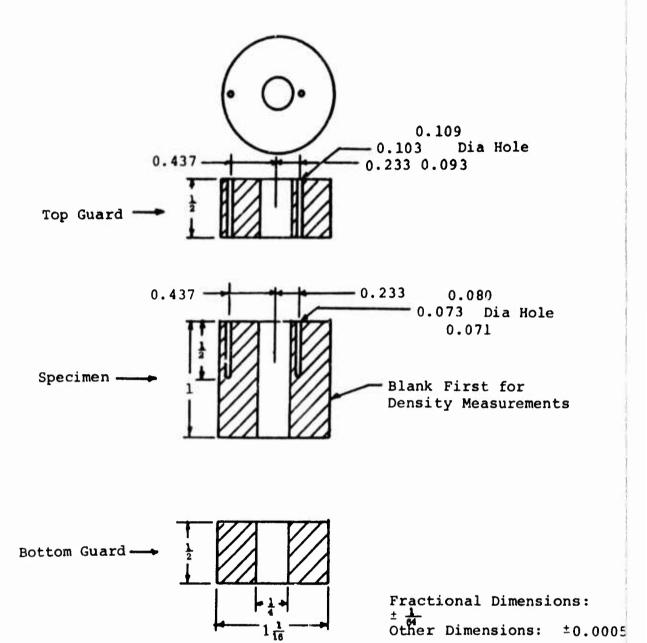
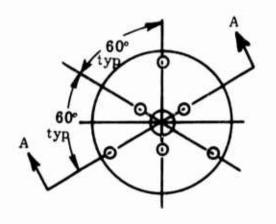


Figure 4. 1.06 Diameter Thermal Conductivity Specimen for Radial Inflow Apparatus



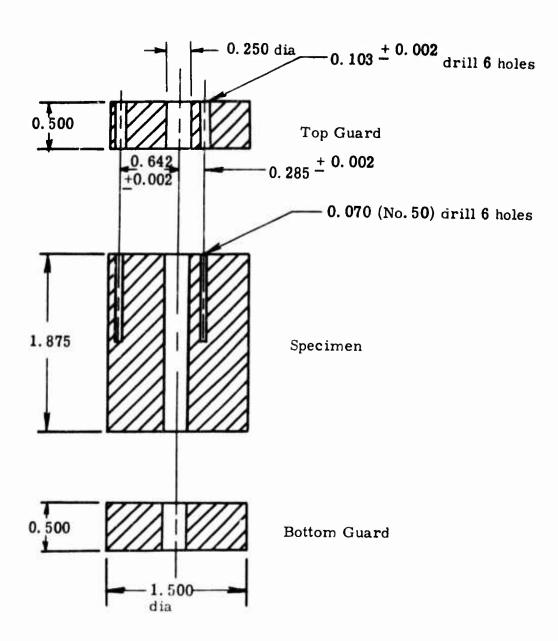
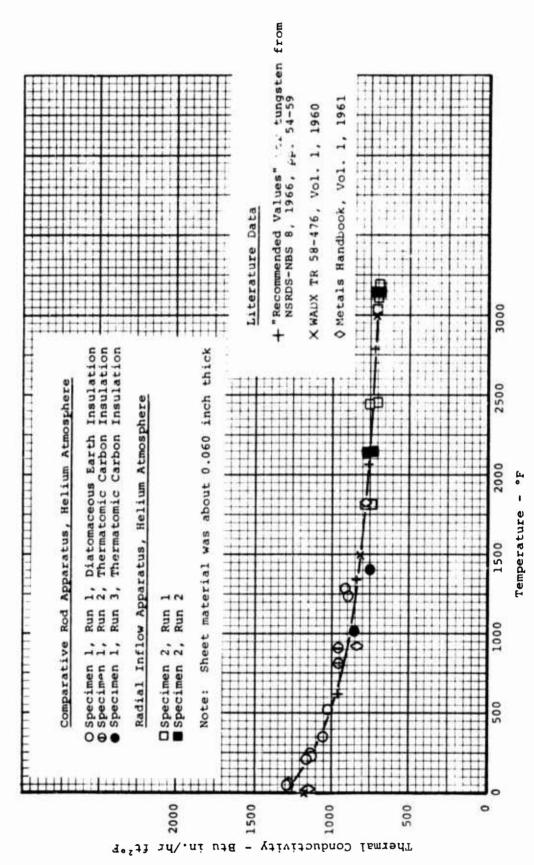
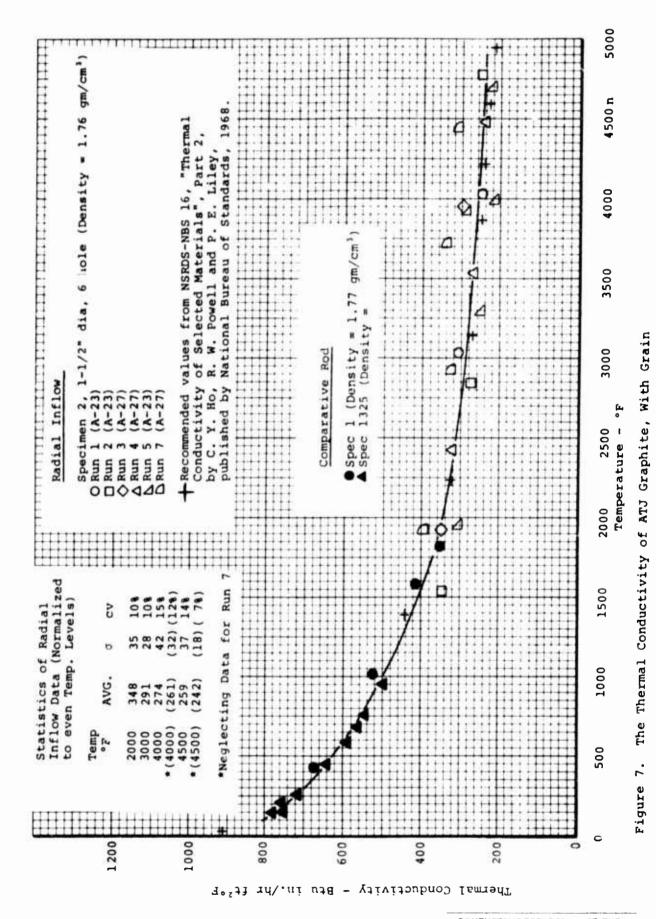


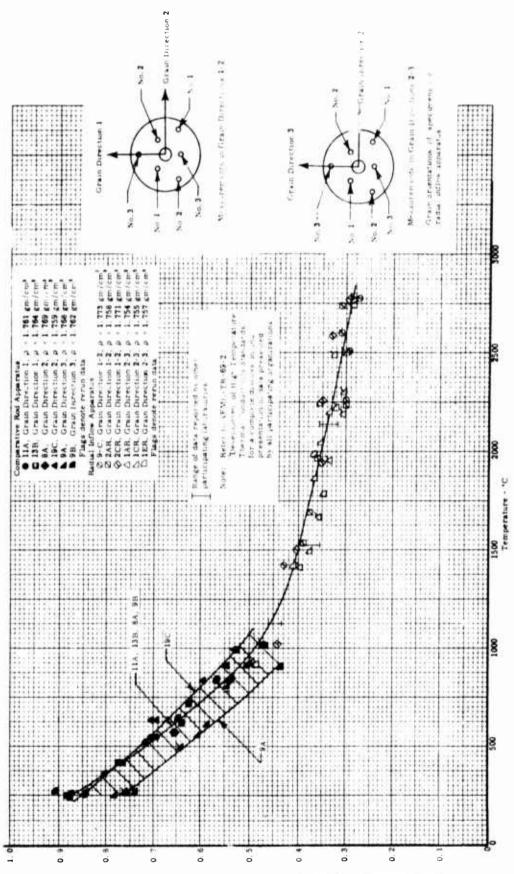
Figure 5. Dimensions of 1.50 Inch Diameter Specimen and Guards Used for Radial Inflow Thermal Conductivity Measurements



The Thermal Conductivity of Tungsten Sheet Parallel to the Plane of the Sheet Figure 6.



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Figure 8. The Thermal Conductivity of AXM-5QI Graphite

APPENDIX II

MECHANICAL PROPERTIES SORI

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MECHANICAL PROPERTIES OF PYROLYTIC GRAPHITE AND CODEPOSITED SIC/PG WITH VARIOUS WEIGHT PERCENTAGES OF SILICON-CARBIDE

FINAL REPORT

to

ATLANTIC RESEARCH CORPORATION Alexandria, Virginia

Purchase Order 78119

Southern Research Institute 2000 Ninth Avenue South Birmingham, Alabama 35205 June, 1973

Project 2931-II-F

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Mechanical Properties of Pyrolytic Graphite and Codeposited SiC/PG with Various Weight Percentages of Silicon-Carbide

INTRODUCTION

This is the final report covering work performed under Atlantic Research Purchase Order 91138 (FO4611-73-C-0012). The tensile and compressive properties were determined for Pyrolytic Graphite and Codeposited SiC/PG with various weight percentages of silicon carbide. The test matrix for the evaluations is presented in Table 1.

The thermal properties of these materials are reported in SoRI Report EAS-73-031 (January, 1973).

MATERIAL

The material was furnished by Atlantic Research Corporation primarily in the form of cylinders of various lengths up to 9 inches. The pyrolytic graphite was deposited on the 2 inch I.D. of AGSR graphite cylinders and the codeposited SiC/PG was deposited on the 2 inch I.D. of ATJ graphite cylinders. The thicknesses of the deposited layers varied from cylinder to cylinder and along each cylinder length. The pyrolytic graphite layers on the AGSR cylinders were not as thick as the codeposited SiC/PG on the ATJ and as a result, representative coupon specimens could not be obtained for the PG material.

In an attempt to supply thicker samples of PG, Atlantic Research prepared 6 strips of PG approximately 13 inches long \times 1-3/4-inch wide \times various thicknesses. Although thicker, the strips were warped and we were unable to obtain straight specimens with sufficient thickness.

In an effort to obtain some tensile information on the Atlantic Research PG material, two small pieces (033-25), with nominal dimensions of 3-1/4-inches x 2 inches x approximately 0.1 inch thickness were utilized to obtain four subsize tensile specimens. (See Figure 1.)

From the materials supplied, the following cylinders were selected for evaluation:

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Material	Nominal	Number and Type of
Designation	Composition	Specimen obtained
033-25 ^a C- 1 C- 3 C-39 C-43 C-55 C-55 C-51 C-16 C-18 C-45 C-51 C-49 C-23 C-21	PG 20% SiC/PG 20% SiC/PG 20% SiC/PG 20% SiC/PG 20% SiC/PG 8% SiC/PG 8% SiC/PG 20% SiC/PG PG PG PG 20% SiC/PG	4 Coupon tensiles 1 Cylinder tensile 1 Cylinder tensile 5 Coupon tensiles 5 Coupon tensiles 5 Coupon tensiles 5 Coupon tensiles 6 Coupon tensiles 7 Coupon tensiles 7 Cylinder compression 8 Cylinder compression 9 Cylinder compression 1 Cylinder compression
C-33	20% SiC/PG	5 Coupon compressions
C-59	8% SiC/PG	5 Coupon compressions
C-30	PG	5 "C" Coupon tensiles

APPARATUS AND PROCEDURES

Tension

The tensile evaluations were performed in a gas-bearing tensile facility. The tensile machine utilizes gas-bearing universals in the load linkage to prevent the introduction of unknown bending moments into the load train from crosshead motion and to allow monitoring of the straightness of the load train. The primary components of a facility are the load frame, the mechanical drive system, the gas-bearing universals, a 5500°F furnace, optical strain analyzers, and associated instrumentation for measurement of load and strain. The load capacity is 15,000 pounds.

^aFlate plate

bSpecimen was initially evaluated in tension

The load frame and mechanical drive system are similar to those of many good facilities. The upper crosshead is positioned by a small electrical motor connected to a precision screw jack. This crosshead is stationary during loading and is moved only when assembling the load train. The lower crosshead is used to apply the load to the specimen through a precision screw jack, which is chain-driven by a variable speed motor and gear reducer.

The unique feature of the facility that distinguishes it from other good facilities is the use of gas-bearing universals in the specimen load train to eliminate bending moments and hence provide true uniaxial tensile loading of the specimen.

Strain measurements were made using optical strain analyzers. These analyzers track strain targets attached to the specimen and permit an automated continuous readout of strain. A complete tensile facility is described in Appendix A.

Initially, the evaluations were to be conducted on cylindrical specimens for both tension and compression in the ab direction. Two cylindrical tensile specimens (C-1 and C-3) were prepared to the design shown in Figure 2 to check out the feasibility of evaluating cylindrical tube specimens. The first tensile evaluation (C-1) at 70°F resulted in a specimen headfailure at a relatively low stress level. Since the gage section wall thickness was already 0.030 inch, it seemed fruitless to attempt specimen redesign or modification. The decision was made to attempt to use coupon specimens designed as shown in Figure 3, and to modify the remaining cylinder specimen (C-3) so that it could be evaluated to gage failure at 70°F. Specimen C-3 was modified by machining away the ATJ substrate, replacing the ATJ with aluminum sleeves epoxy bonded to the codeposited SiC/PG, and then remachining the aluminum sleeves with the specimen shoulder profile shown in Figure 2.

The coupon tensile specimen design proved successful and modified C-3 cylinder specimen was only partially successful. Therefore, the coupon tensile specimen design was used for the "ab" tensile evaluations of the cylinders.

The "c" direction tensile evaluations at 70°F were also conducted in the gas-bearing tensile facility. Due to the thickness of the material, strain measurements were not possible and strength only was determined when the strength of the test material was less than that of the epoxy bonding used to transfer the load to the specimen. The specimen assembly is shown in Figure 4.

Compression

The compressive evaluations were performed in a gas-bearing compressive facility. The facility is similar in operation to the tensile facility, except the gas-bearing universals are used only to initially precision align the specimen load train and are not in operation during the actual evaluation. The compressive facility is described in Appendix B. The coupon compressive specimen used in the evaluations is shown in Figure 5. To evaluate this specimen in compression required laterally supporting the specimen with low friction inserts to increase the stability. This is a common technique in the evaluation of thin sheets and has been used successfully at SoRI on many past programs. confirm that the coupons yielded representative data, six total cylinder specimens were machined to the design shown in Figure 6 and evaluated in compression. As discussed later, good agreement was obtained between the coupon specimens and these total cylinder specimens.

DATA AND RESULTS

Tension - "ab" Direction

The results of the tensile evaluations of the PG and SiC/PG in the "ab" direction are presented in Table 2 and Figures 7 through 12. Composite plots of the tensile stress-strain curves for 16% SiC/PG, 20% SiC/PG, and 27% SiC/PG are shown in Figures 13, 14, and 15, respectively. Individual stress-strain curves are presented in Appendix C.

The "ab" tensile strength versus temperature for various percentages of SiC are shown in Figure 7. The strengths of the codeposited SiC/PG, for 70°F to 3000°F, were significantly increased by the increasing weight percentage of SiC. The 20 to 27% SiC/PG material exhibited strengths 2 to 3 times greater than the Atlantic Research pure PG (ARC-PG). The 70°F strengths are shown as a function of percentage SiC in Figure 8. The increase in 70°F strengths appeared to be approximately linear with increasing SiC percentage between 10 and 30% SiC.

The strengths of material containing above 20% SiC peaked at about 3000°F, but the material containing 13 and 16% attained peak strengths at about 4000°F. This indicates that the strengthening effects of the SiC are probably maximum at about 3000°F and above 3000°F the effects decrease rapidly to 4500°F. Figures 8, 9, and 10 illustrate this observation. In Figures 8 and 9, the 70°F and 3000°F tensile strengths are shown to be a strong, almost linear, function of SiC content. At 4500°F (see Figure 10), the strengthing effect of the SiC has diminished significantly, suggesting very little correlation with SiC content.

The "ab" tensile elastic moduli versus temperature for various percentages of SiC/PG are presented in Figure 11. As with the strength, the elastic modulus was an increasing function of SiC content. At 70°F, the moduli of ARC-PG was about 3.5 x 10°psi and for 27% SiC/PG was about 5.7 x 10°psi. The stiffening effect of the SiC was observed in all the evaluations to above 4000°F. At 4500°F, the stiffening effect was diminished but could still be observed in the data. The moduli values for all the various percentages of SiC/PG peaked at 3000°F.

The "ab" tensile strains-to-failure versus temperature for the various percentages of SiC/PG are shown in Figure 12. The data indicate an increase in strains-to-failure with increasing SiC content from 70°F to above 4000°F. The data were not as ordered as the strength and modulus data but the 20 to 27% SiC content specimens exhibited higher total strains than the 13 to 16% SiC content specimens. At 4500°F, the results were mixed.

A composite of the tensile stress-strain curves for 16% SiC/PG (Cylinder C-55) are shown in Figure 13. The stress-strain response was linear to fracture at 70°F and 3000°F but became slightly bilinear between 3000°F and 4000°F. Bilinear response was recorded at 4500°F. The stress-strain response of the 20% SiC/PG (Cylinder C-35) was similar as shown in Figure 14. The stress-strain response of the 27% SiC/PG (Cylinder C-39) was slightly bilinear at 70°F and became increasingly bilinear with increasing temperature (See Figure 15).

Tension - "c" Direction

The results of the "c" direction tensile strength evaluations at 70°F are presented in Table 3. Tensile failures of specimen materials were obtained only in the ARC-PG specimens. In the SiC/PG specimens, tensile failures occurred in the epoxy bond (see Figure 4). The average "c" direction tensile strength of the ARC-PG at 70°F was 792 psi and the strengths of the SiC/PG specimens were greater than 3000 psi. Therefore, the additions of SiC to PG resulted in a significant increase in "c" direction tensile strength.

Compression

The results of the compressive evaluations of the PG and SiC/PG are presented in Table 4. The compressive moduli versus temperature are presented in Figure 16 and composite plots of the compressive stress-strain curves for 13% SiC/PG, 16% SiC/PG, and 25% SiC/PG materials are presented in Figures 17, 18, and 19. Individual stress-strain curves are presented in Appendix D.

The effect of SiC content on the compressive elastic modulus was approximately the same as in tension; increase in modulus with increasing SiC content (see Figure 16). Although the compressive runs at elevated temperatures were conducted at only 3000°F and 4500°F, the moduli apparently peaked at 3000°F; the same as the tensile moduli. The stiffening effects of the SiC content were diminished significantly at 4500°F.

As mentioned earlier, the majority of the compressive evaluations were performed on coupon specimens (see Figure 5) using lateral supports to increase stability. To confirm that the coupon specimens gave representative compressive data, several total cylinder specimens were evaluated at 70°F for comparison. Good agreement between the total cylinders and coupon specimens was obtained as shown in Figures 18 and In these figures, the compressive stress-strain response of the total cylinder specimen; are shown as dashed curves. The coupon specimens generally gave excellent stress-strain data at strains up to 0.010 to 0.020 in./in. In this strain range, most stress-strain records showed a sudden increase in slope (see Appendix D) indicating that the lateral supports were probably beginning to support some of the compressive load. Stress-strain data after the sudden increase in curve slope were disregarded.

For comparison, the compressive stress-strain curves for the total cylinders of ARC-PG (Cylinders C-16 and C-18) are included in the composite plot in Figures 17, 18, and 19. The addition of SiC changed the character of the stress-strain curves from linear to bilinear. Strength, stiffness, and strain-to-failure increased with increasing (13% to 25%) SiC content at 70°F and 3000°F. At 4500°F, the stress-strain responses appeared to be independent of SiC content.

CONCLUSIONS

The addition of SiC to PG increased the strength, stiffness, and strains-to-failure in both tension and compression over the temperature range 70°F to 3000°F. The increase in these properties appeared to be almost linear with increasing

SiC content between 10 and 30%. Above 3000°F, the strengthening effect of the SiC began to decrease and was significantly diminished at 4500°F.

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Mechanics Division

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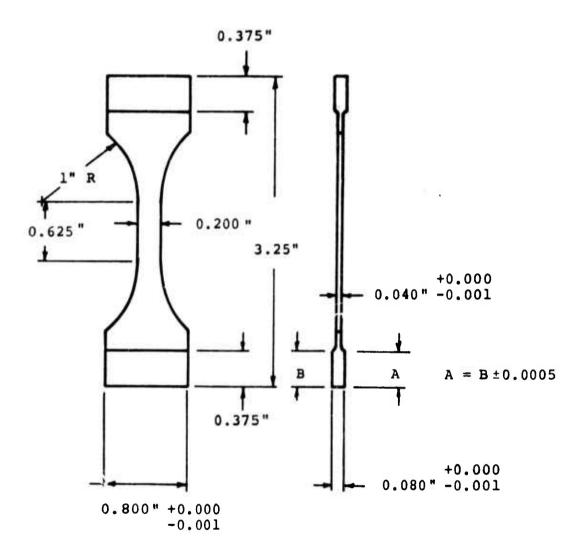


Figure 1. Subsize Tensile Specimen for PG Flat Plate

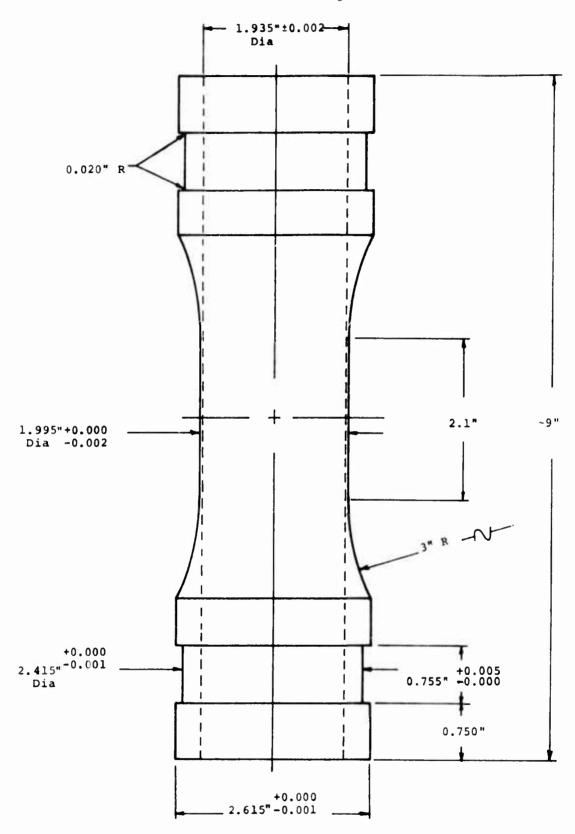


Figure 2. Axial Cylindrical Tensile Specimen for Codeposited PG/SiC on ATJ $\,$

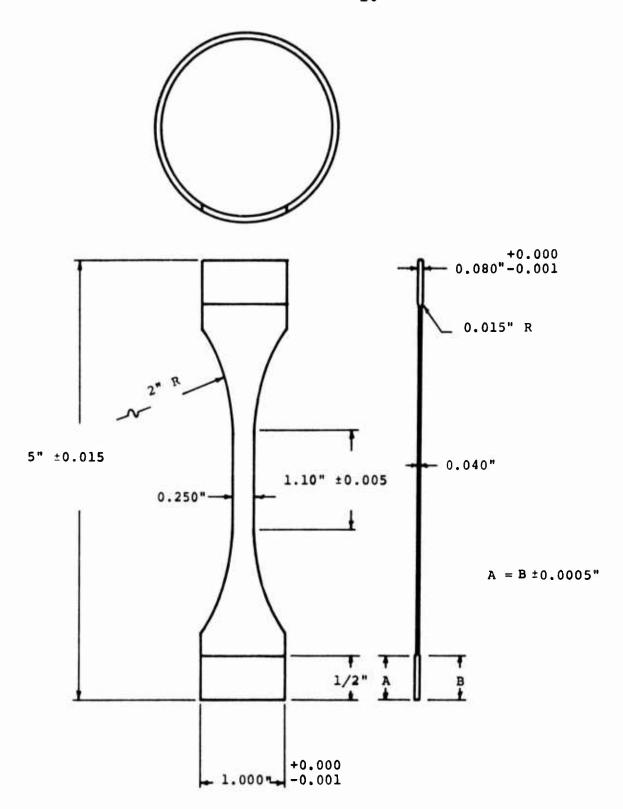


Figure 3. Coupon Tensile Specimen for Evaluation of Codeposited PG/Sic Cylinders in the ab Direction

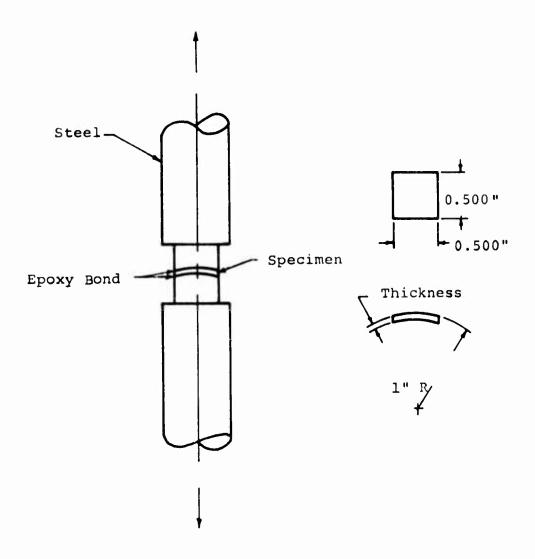


Figure 4. Specimen Assembly for Evaluation of PG and Codeposited SiC/PG Cylinders in the "c" Direction

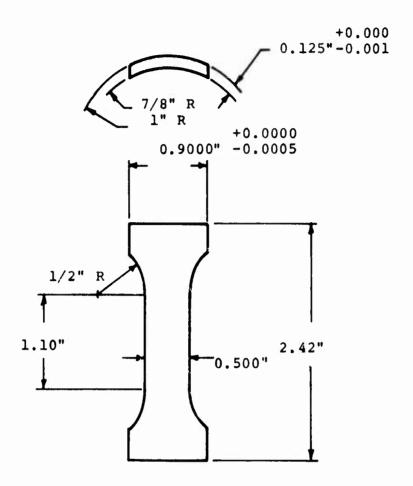
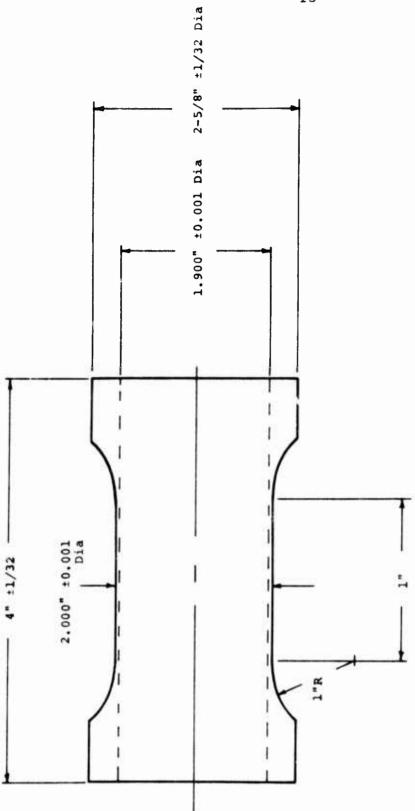


Figure 5. Compressive Specimen for Codeposited PG and SiC Cylinders



Cylindrical Compressive Specimen for the Evaluation of Codeposited PG/SIC in the ab direction Figure 6.

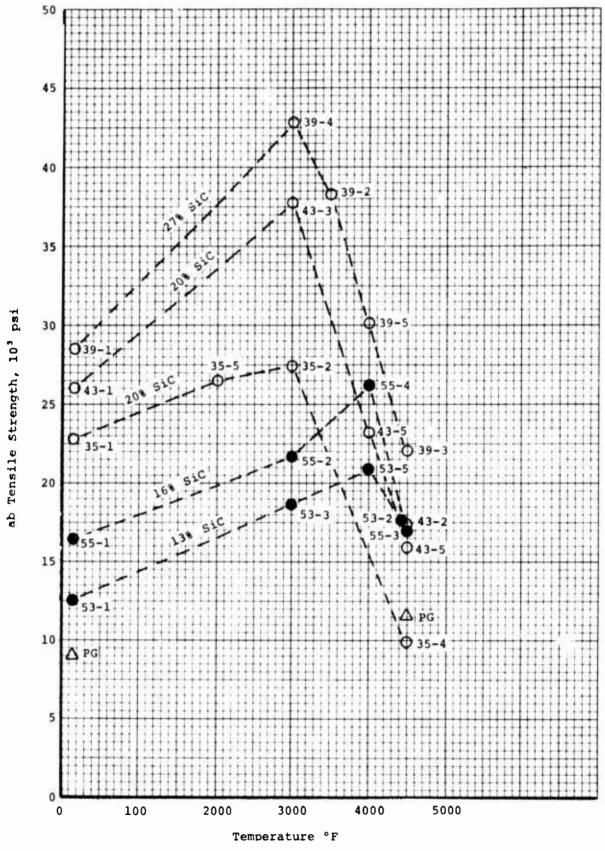


Figure 7. Tensile Strength Versus Temperature for Codeposited PG/SIC in the ab Direction

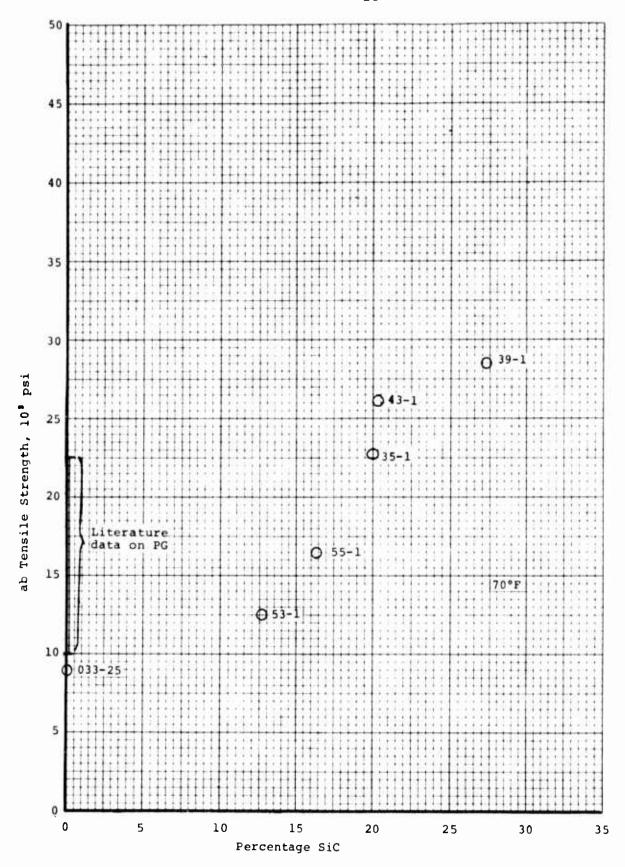


Figure 8. ab Tensile Strength at 70°F Versus Percentage SiC for Codeposited Sic/PG Cylinders

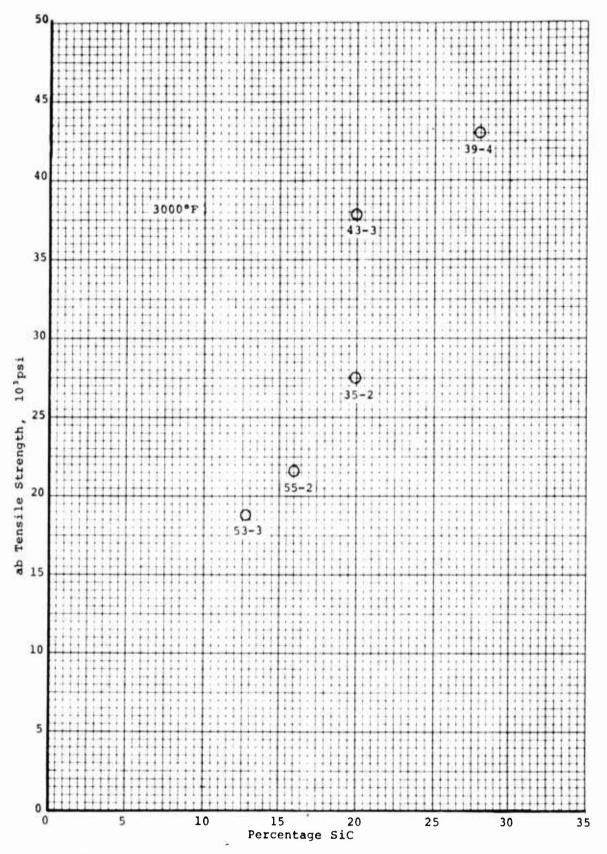


Figure 9. ab Tensile Strength at 3000°F versus Percentage SiC for Codeposited SiC/PG Cylinders

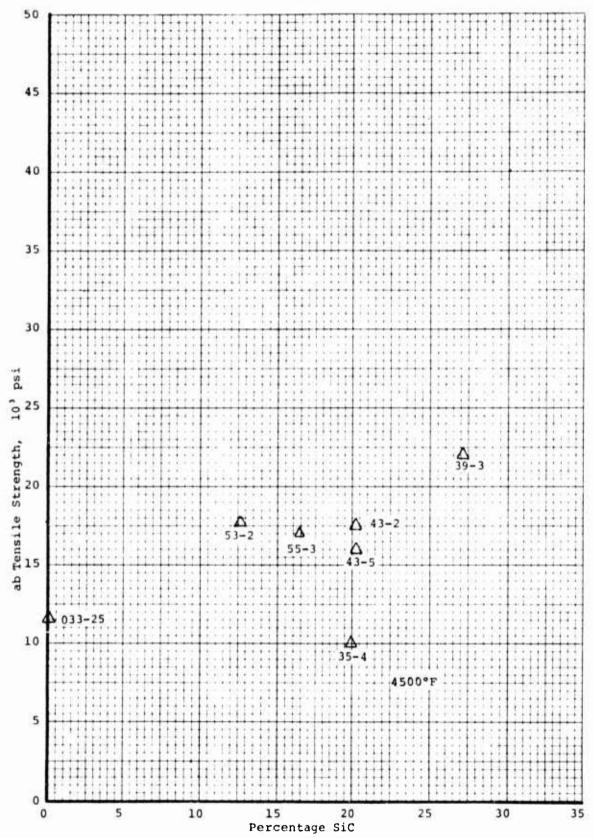
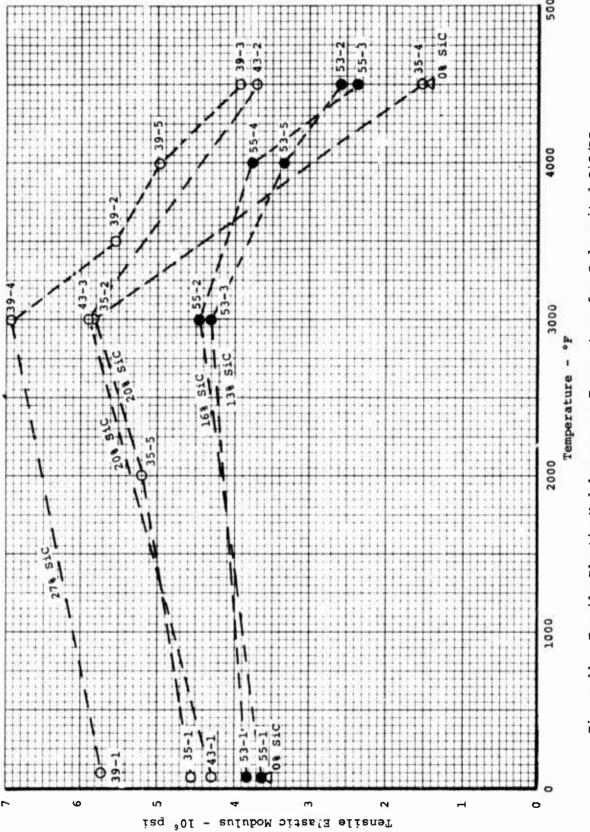


Figure 10. ab Tensile Strength at 4500°F Versus Percentage SiC for Codeposited SiC/PG Cylinders



Tensile Elastic Modulus versus Temperature for Codeposited SiC/PG in the ab Direction Figure 11.

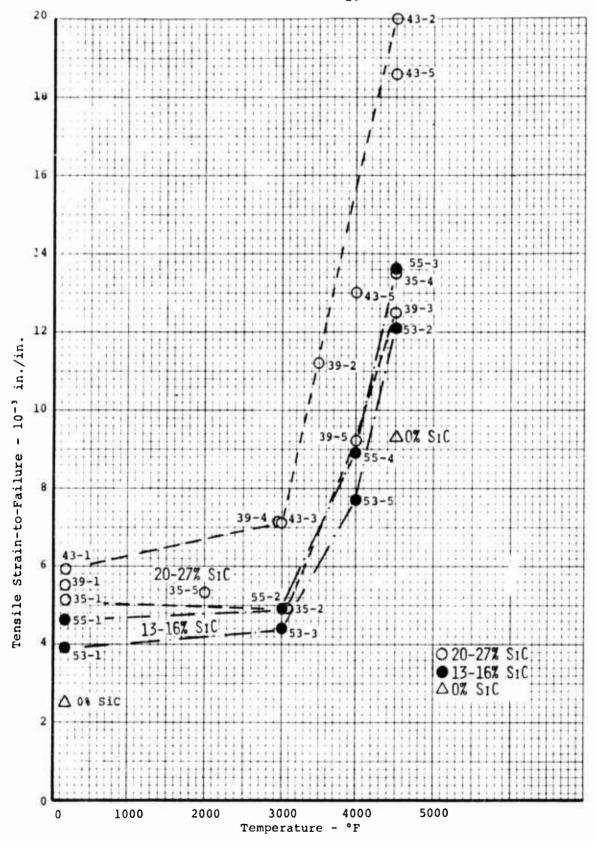


Figure 12. Tensile Strain-to-Failure versus Temperature for Codeposited SiC/PG in the ab Direction

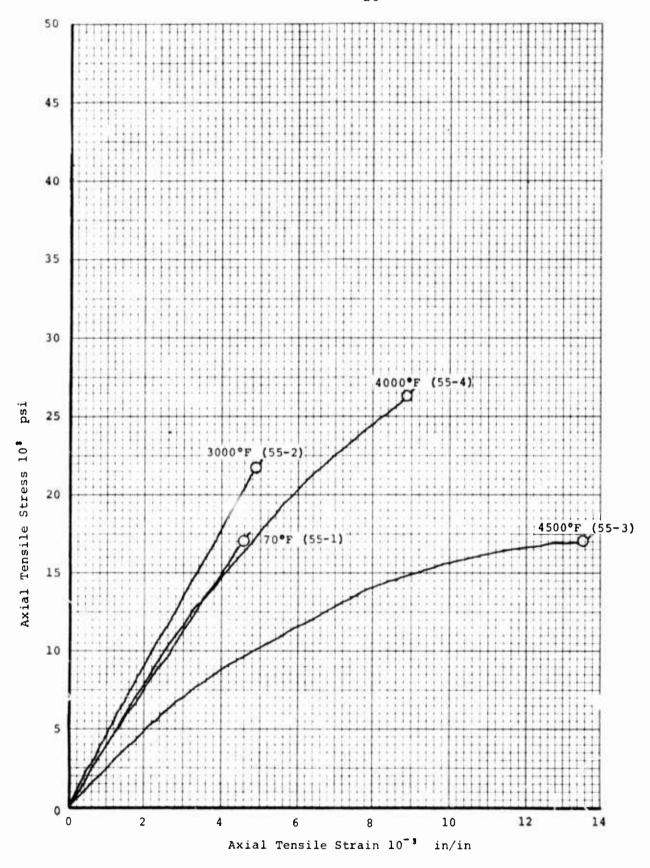


Figure 13. Tensile Stress-Strain Curves For Codeposited 16% SiC/PG (Cylinder C-55)

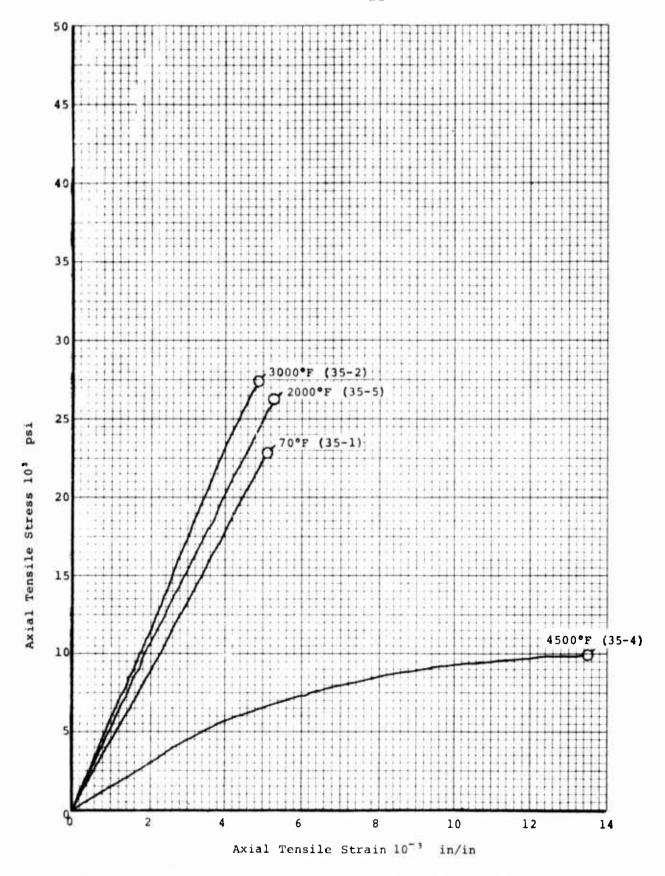


Figure 14. Tensile Stress-Strain Curves for Codeposited 20% SiC/PG (Cylinder C-35)

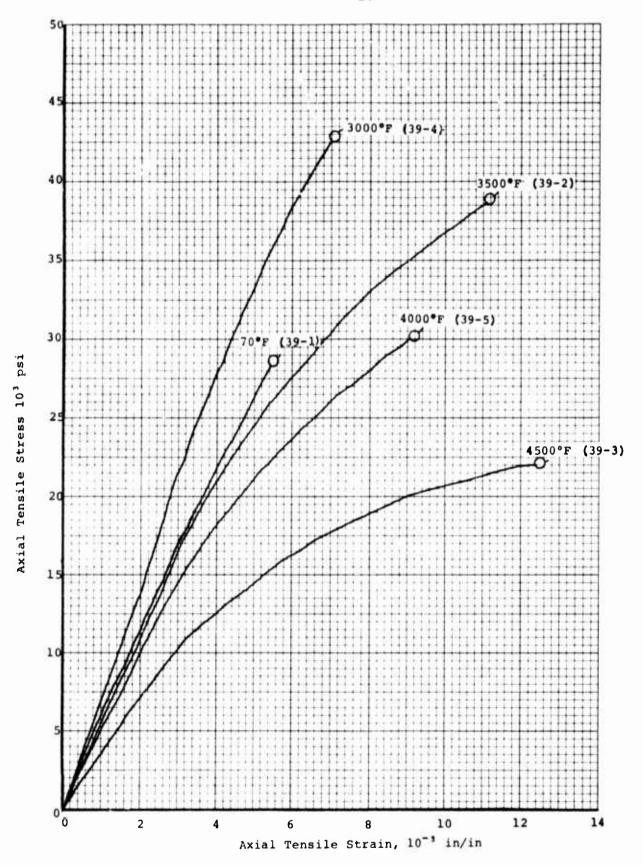
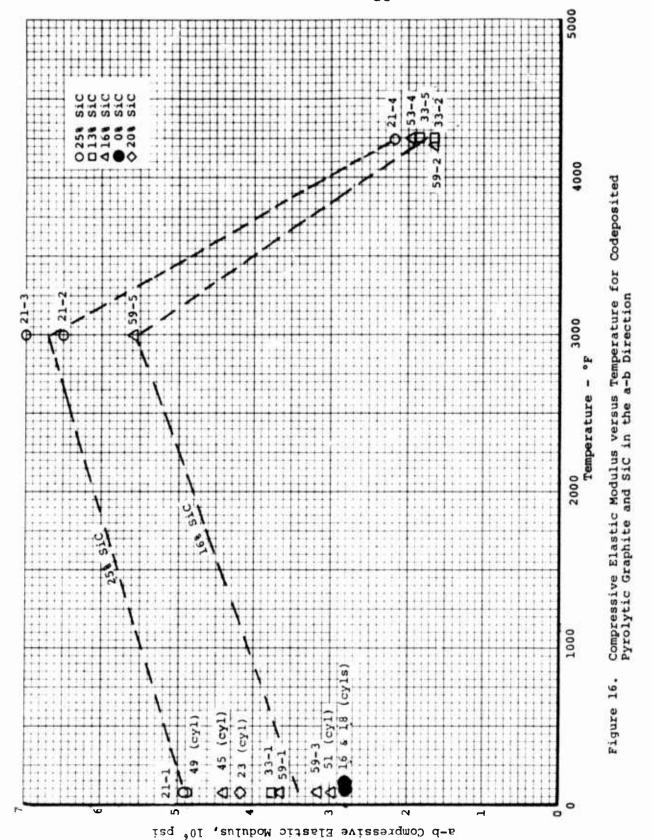
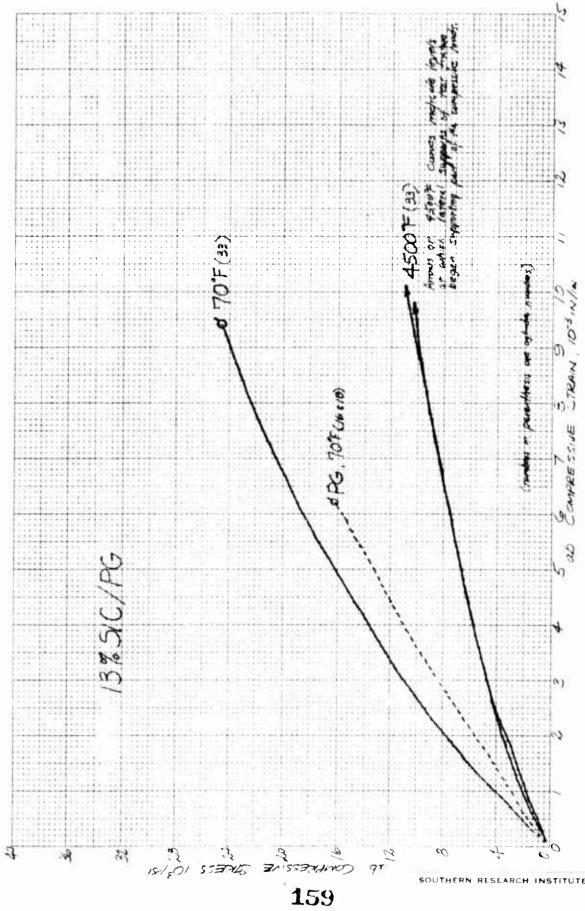


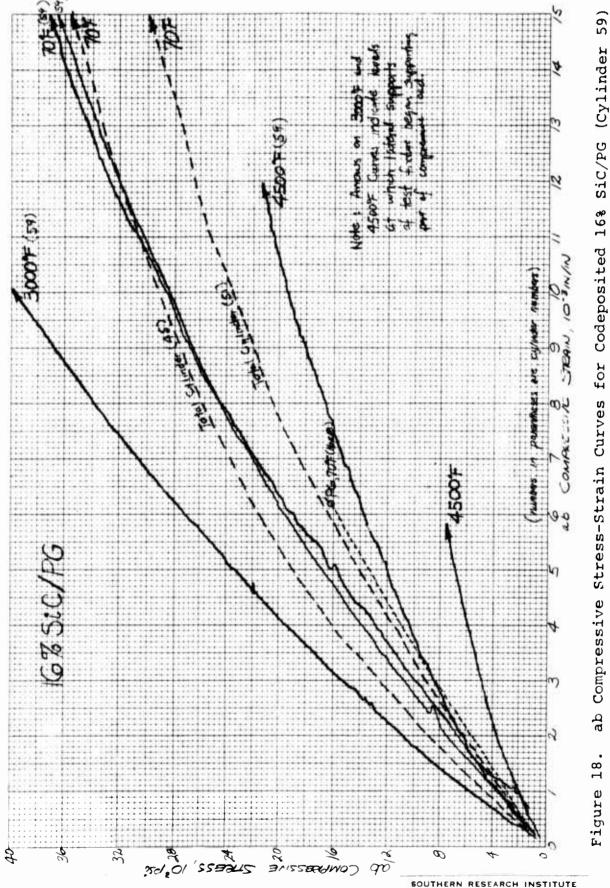
Figure 15. ab Tensile Stress-Strain Curves for Codeposited 27% SiC/PG (Cylinder C-39)



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ab Compressive Stress-Strain Curves for Codeposited 13% SiC/PG (Cylinder 33) Figure 17.



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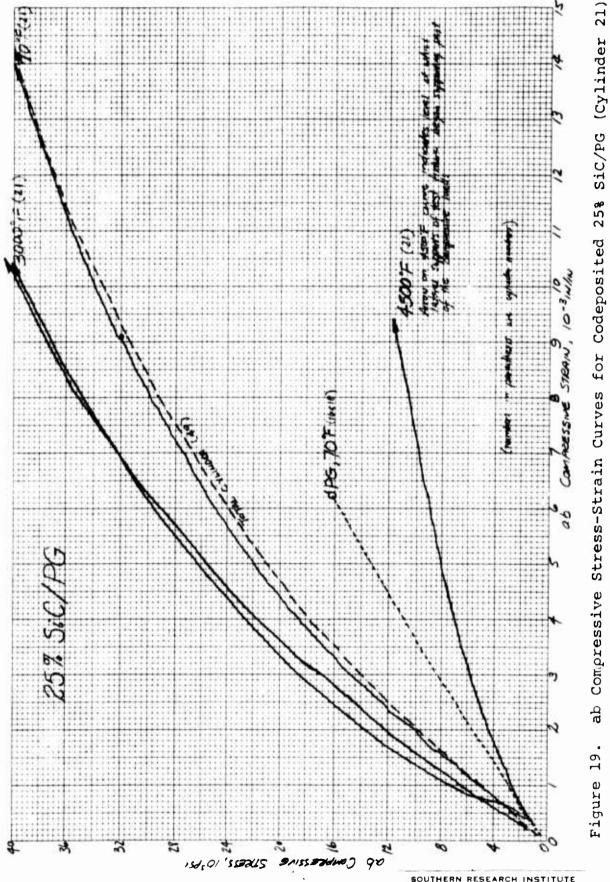


TABLE 1

Mechanical Property Test Matrix for Evaluation of PG and Codeposited SiC/PG

4500	2(1) 2(2) 2(4)	1 [1	2 (0) 2 (2) 2 (3)
4000	- (2) - (2)	1 1 1	1 1 1
Temperature °F 3000 3500	- (1)	1 1 1	1 1 1
Tempera 3000	- (2) 2 (3)	T 1 T	- -(1) -(2)
2000	- (1)	1 1 1	1 1 1
70	2(1) ^a 2(2) 2(5)	3(3) 3(3) 4(4)	2(2) 2(3) 2(5)
Material	PG 8% SiC/PG 20% SiC/PG	PG 8% SiC/PG 20% SiC/PG	PG 8% SiC/PG 20% SiC/PG
Orientation	ab	U	ab
Property	Tension		Compression

anumbers in parentheses are the actual evaluations performed

Tensile Data for PG and Codeposited SiC/PG Cylinders

Remarks	Broken in machining Broken in load train assembly						Poisson's Ratio (ab/ab) = 0.14	Poisson's Ratio (ab/ab) = 0.14
Nominal Weight	0000	co co co co co	00 00 00 00	50 50 50 50	00000	20 20 20 20 20	20	20
Total Unit Axial Strain in./in.	0.0025	0.0039 0.0044 0.0077 0.0121	0.0046 0.0049 0.0089 0.0136	0.0051 0.0053 0.0049 0.0135	0.0059 0.0071 -0.013 0.0204 0.0186	0.0055 0.0071 0.0112 0.0092 0.0125	>0.0019	>0.0021
Initial Elastic Modulus 10 psi	3.6	3.82 4.30 3.32 2.58	3.62 4.46 3.77 2.36	4.55 5.20 5.82 1.51	5.90b 3.72	5.72 6.95 5.52 4.98 3.55	5.25	6.21
Ultimate Strength psi	8,960	12,600 18,600 20,800 17,600	16,400 21,600 26,200 16,900	22,800 26,500 27,400 9,880	26,000 37,800 23,200 17,400 15,800	28,500 42,900 38,300 30,100 22,000	> 9,640	>12,000
Bulk Density gm/cm	2.18 2.19 2.20 2.20	2.38	2.35 2.37 2.37	2.45	2.41	2.47 2.46 2.48 2.50	!	
Specimen Number	033-25-T1a 033-25-T2a 033-25-T3a 033-25-T4a	C-53-1T C-53-3T C-53-5T C-53-2T	C-55-1T C-55-2T C-55-4T C-55-3T	C-35-1T C-35-5T C-35-2T C-35-4T	C-43-1T C-43-1T C-43-4T C-43-2T C-43-5T	C-39-1T C-39-4T C-39-2T C-39-5T C-39-3T	C-1-TC	C-3-IC
Stress Rate psi/sec	10,000	10,000	10,000	10,000	10,000	10,000	10,000	10,000
Temp • F	4500	3000 4000 4500	3000 4000 4500	2000 3000 4500	3000 4000 4500 4500	3000 3500 4000 4500	70	20
Loading Direction	qe ,	q	qe	qe	ф	Qe	q a	
Meas Wt	0	13	16	20	20	27	1	;

aFlat plate specimens

Crotal cylinder specimens

^bInitial portion of stress-strain curve was erratic

Table 3 Tensile Strength Data for PG and Codeposited Sic/PG at 70° F in the "C" Direction

Measured Weight Percentage SiC	Specimen No	Ultimate Tensile Strength, psi	Nominal Weight Percentage SiC
0	C-30-1 C-30-2 C-30-3 C-30-4	_ a 780 725 870 792	0 0 0
13	C-53-1 C-53-2 C-53-3 C-53-4	_ a >3000b >3040b >2950	8 8 8
27	C-39-1 C-39-2 C-39-3 C-39-4	>4300 ^b >3040 ^b >3250 ^b >3300 ^b	20 20 20 20

abroken during assembly
bepoxy bond failure; specimen did not fail

Table 4
Compressive Data for PG and Codeposited SiC/PG

Remarks	Poisson's Ratio = -0.09	Poisson's Ratio = -0.08 (ab/ab)			Poisson's Ratio = 0.13	Poisson's Ratio = 0.14 (ab/ab)				Poisson's Ratio = 0.09	Poisson's Ratio = 0.15	(ab/ab)		
Nominal Weight	0	0	20	20	20	60	00 00	œ	00 00	20	25	20	20 20	20
Total Unit Axial Strain in./in.	0.0062	0.0062	0.0094	0.0097 ^b	0.026	0.023	-0.020 ^b	0.0101b	0.012 ^b 0.0059 ^b	0.019	0.0220	0.0167b	0.0105b 0.0118b	Q*600°0
Initial Elastic Modulus 10 psi	2.78	2.78	3.74	1.63	4.41	2.98	3.64	5.58	1.63	4.19	4.91	4.92	6.5	2.18
Ultimate Strength psi	16,000	16,100	25,000	10,400b	46,500	36,600	43,200b	40,000b	10,700b 7,360b	32,700	51,000	43,200b	40,500b	11,800b
Bulk Density gm/cm ³	1	ı	2.34	2.33	1	ı	2.34	2.35	2.34	ı	,	2.44	2.43	2.43
Specimen	C-16ª	C-18ª	C-33-1C	C-33-2C C-33-5C	C-45ª	C-51ª	C-59-1C C-59-3C	C-59-5C	C-59-2C C-59-4C	C-23ª	C-49ª	C-21-1C	C-21-2C C-21-3C	C-21-4C
Stress Rate Psi/sec	10,000		10,000	10,000	10,000		10,000	10,000	10,000	10,000	10,000	10,000	10,000	10,000
Temp • F	70		70	4500	70		70	3000	4500	70	70	70	3000	4500
Load Direction	ab		ab		qe					ab	qe			
Meas Wt %	•		13		16					20	25			

Total cylinder specimen Stress and strain level at which lateral supports of the test fixture began supporting part of the load

APPENDICES

- A Ultimate Strength, Elastic Modulus and Poisson's Ratio to 5500°F in Tension
- B Ultimate Strength, Elastic Modulus and Poisson's Ratio to 5500°F '.n Compression
- C Tensile Stress-Strain Curves for PG and Codeposited Sic/PG in the AB Direction
- D Compressive Stress-Strain Curves for PG and Codeposited Sic/PG in the AB Direction

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APPENDIX A

ULTIMATE STRENGTH, ELASTIC MODULUS AND POISSON'S RATIO TO 5500°F IN TENSION

A typical tensile facility is shown in the photograph in Figure 1 and in the schematic in Figure 2. The primary components are the gas-bearings, the load frame, the mechanical drive system, the 5500°F furnace, the optical strain analyzers and associated instrumentation for measurement of load and strain. The load capacity is 15,000 pounds.

The load frame and mechanical drive system are similar to those of many good facilities. The upper crosshead is positioned by a small electric motor connected to a precision screw jack. This crosshead is stationary during loading and is moved only when assembling the load train. The lower crosshead is used to apply the load to the specimen through a precision screw jack chain driven by a variable speed motor and gear reducer.

Nonuniaxial loading, and therefore bending stresses, may be introduced in tensile specimens not only from (1) misalignment of the load train at the attachment to the crossheads, but also from (2) eccentricity within the load train, (3) unbalance of the load train and (4) external forces applied to the load train by such items as electrical leads and clip-on extensometers. Although the bending moments from some of these sources may seem relatively slight, the resulting stress distortions are quite significant in the evaluation of the extremely sensitive brittle materials. Now consider each individually.

To confirm that the gas-bearings had eliminated nonuniaxial loading at the point of attachment of the load train to the crossheads, the frictional moment was determined at a load of 5000 pounds by measuring the torque required to produce initial motion within the system with the bearings in operation. This torque was found to be a maximum of 6.6×10^{-3} inch-pounds. The equation

$$M_{O} = \frac{2\mu P}{3} \qquad \left[\frac{R_{2}^{3} - R_{1}^{3}}{R_{2}^{2} - R_{1}^{2}} \right]$$
 (1)

was then applied to the system to calculate the kinetic friction where M_O was the resisting moment due to kinetic friction and μ represented the coefficient of kinetic friction. The calculated value of μ was then equal to a maximum of only 4.5 x 10^{-7} .

The classic equation

$$S = \frac{Mc}{T}$$
 (2)

was then employed to obtain the stress that could be induced in the specimen due to this bending moment. This value was 0.16 psi, or less than 0.002 percent of the tensile stress produced within a typical graphite specimen. These low values clearly indicate the elimination of problems of bending stress in the pecimen imposed by misalignment at the crosshead attachments, either initially or during loading.

Emphases in the design of the load train were placed on (1) large length-to-diameter ratios at each connection, (2) close sliding fits (less than 0.005 inch) of all mating connections, (3) the elimination of threaded connections, (4) the use of pin connections wherever possible and (5) increasing the size of components to permit precise machining of all mating surfaces. All members were machined true and concentric to within 0.0005 inch, and the entire load train was checked regularly to ensure overall alignment following assembly of the individual members. This process ensures concentricity and no kinks in the system.

The problems of unbalance within the load train and of external forces applied to the load train have been explored and corrected. The entire load train is statically balanced to less than 0.01 inchpound for normal operation.

One configuration of the tensile specimen is shown in Figure 3. This specimen provides a relatively large L/D ratio in the gripping area to ensure good alignment. All surfaces in the gripping area are cylindrical in order to make precision machining easier and repeatable from specimen to specimen. This specimen also has double breakdown radii from the gripping area to the gage section. This double breakdown allows a uniform transition of the stress pattern and reduces the frequency of radius (out of gage) fractures. This specimen provides a uniform gage section which gives a definable volume of material under stress and permits accurate measurements of strain. The flags for the measurement of axial strain are positioned one inch apart so that unit strain is recorded directly. The flag attachment for measurement of lateral strain is positioned between the flags for axial strain; see Figure 4.

A schematic of the precision tensile grip is shown in Figure 5. The design is much like the jaws of a lathe head on the chuck of a drill motor made with precision. Observe from the figure the long surface contact of the mating parts and the close fits to establish precise alignment with the specimen. As the load is applied, the wedges maintain alignment to fracture.

Figure 6 is a sketch of the 5500°F furnace used for tension showing the basic components. The furnace consists of a resistively heated graphite element insulated from a water-cooled shell by thermatomic carbon. The furnace and specimen are purged with helium to provide an inert atmosphere. Ports with visual openings are provided on opposite sides of the furnace as a means of allowing the strain analyzers to view the gage flags on the specimen. Specimen temperatures are determined by optical pyrometer readings taken through another small sight port containing a sapphire window. A calibration curve was established for the loss through the sapphire window, and since the furnace cavity acts essentially as a blackbody, true temperature readings are obtained. Power is supplied to the heating element by means of a 25 KVA variable transformer.

Strain measurement consists of measuring optically the elongation between two flags, or targets, which are mounted on the specimen and separated initially by a predetermined gage length. The travel of the targets is measured by sensing the displacement of the image of the edge of the targets and then electromechanically following the image displacement. The relative travel of the two targets provides the strain. Readout is continuous and automatic on a millivolt recorder. A schematic of the analyzer is shown in Figure 7.

A brief summary of the mechanical motions of the components involved in monitoring the strain is helpful in understanding the detailed performance. A tracking telescope follows the upper target and carries a second telescope mounted on its carriage. The second telescope is capable of independent motion to follow the lower target. The relative displacement between the upper and lower telescope, as strain occurs, defines the strain. The system usually is operated so that the tracking telescope follows the upper target and the strain is monitored by the relative displacement of the aperture rather than the telescope following the lower target. With this procedure the maximum range is the maximum displacement available for the lower aperture, of about 1/8 inch, and the sensitivity is limited by the optics and the noise level of the detector. Using both telescopes, the range is about 3/4 inch.

To provide optical references on the specimens, targets are affixed to the test specimen as mentioned. When the specimen is heated to temperature, the targets are self-luminous and are observed optically. The optics view past the luminous targets into a cooled cavity in the opposite furnace wall. The self-luminous targets are then visible against a dark background. To obtain data below 2000°F, a light beam is directed from behind the flags providing a shadow image for the detection system.

The image of the flowing target is focused through a rotating shutter (chopper) and onto a rectangular aperture. Small slits in the aperture pass a portion of the upper and lower edges of the light beam. A photocell receives the light thus transmitted, and an electronic circuit detects whether the energy passed by the two slits is equal. A servo drives the apertures to let a balanced quantity of light pass through the two slits and thus maintains an optical null.

To obtain lateral strain, a strain analyzer is supported horizontally on the tensile frame to view the diametrical or lateral strain of the specimen. A flag attachment, with the general configuration as shown in Figure 8, was developed to follow and transmit lateral motions of up to a few mils. The three-piece assembly consists of a ring and two rams bearing on the specimen.

Calibrations of the analyzers are performed in various ways including absolute correlations to precision micrometers, strain gage extensometers, and direct plots of stress-strain for reference materials such as steel, plexiglas, magnesium and aluminum. Precision is within ±0.000020 inch.

Instrumentation includes primarily a stress-strain measurement system composed of a 1000-pound SR-4 Baldwin load cell, constant d.c. voltage power supply, two optical strain analyzers, and two X-Y recorders. Specimen temperature is monitored with an optical pyrometer. Stress (load) is measured by a commercial load cell. The cell receives a constant d.c. voltage input from the power supply and transmits a millivolt signal (directly proportional to load) to an X-Y recorder. Simultaneously, the optical strain analyzers measure both the axial and lateral strain and transmit a millivolt signal (proportional to strain) to the X-Y recorders. Thus, continuous plots of stress-axial strain and axial strain-lateral strain are recorded simultaneously.

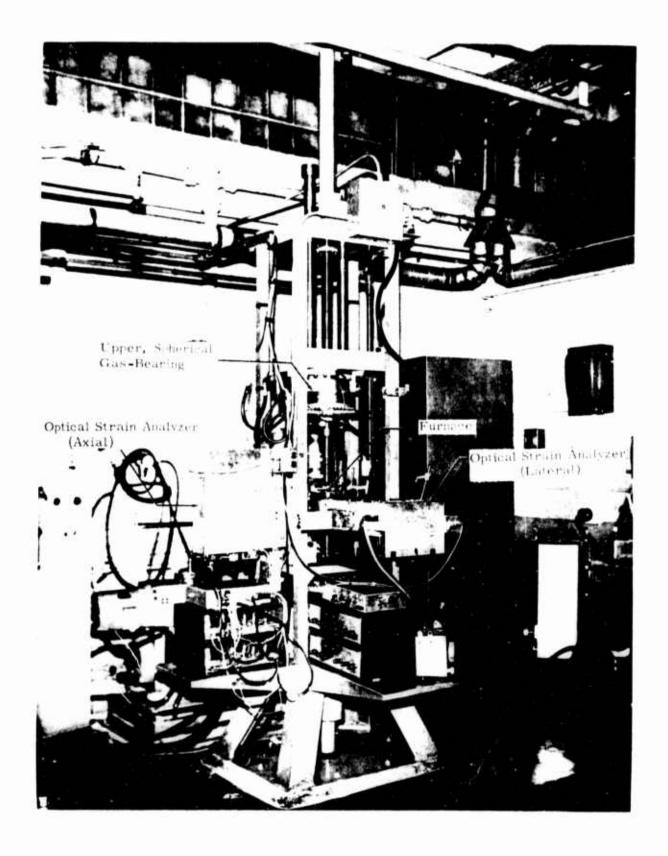


Figure 1. Picture of a Tensile Stress-Strain Facility

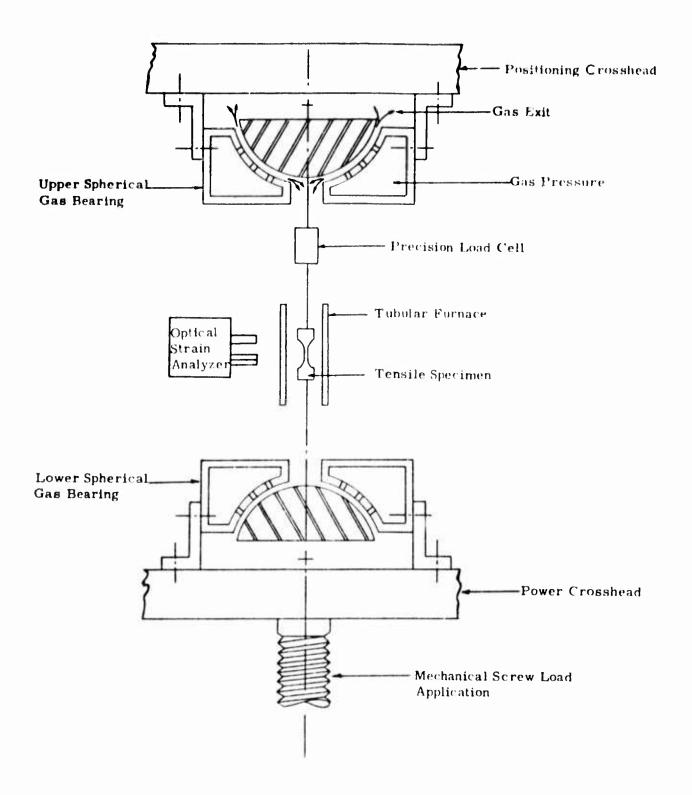


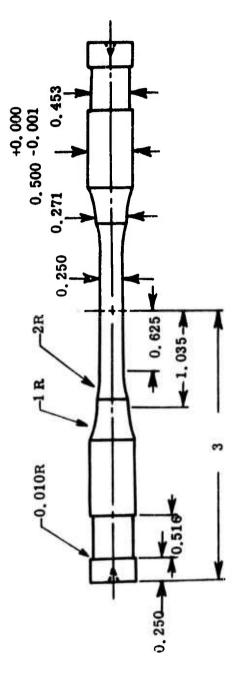
Figure 2. Schematic Arrangement of Gas-Bearing Universals, Specimen and Load Train

All Diameters True and Concentric to Within 0,0005" મ. છ. છ. 4. ષ.

Do Not Undercut Radii At Tangent Points

Both Ends Flat and Perpendicular to and to Within 0,0005"

All Dimensions are in Inches
Tolerances are \$\div 0.001\$ on Diameters
\$\div 0.005\$ on Lengths
\$\div \frac{1}{64}\$ on Fractions



Tensile Specimen Configuration Figure 3.

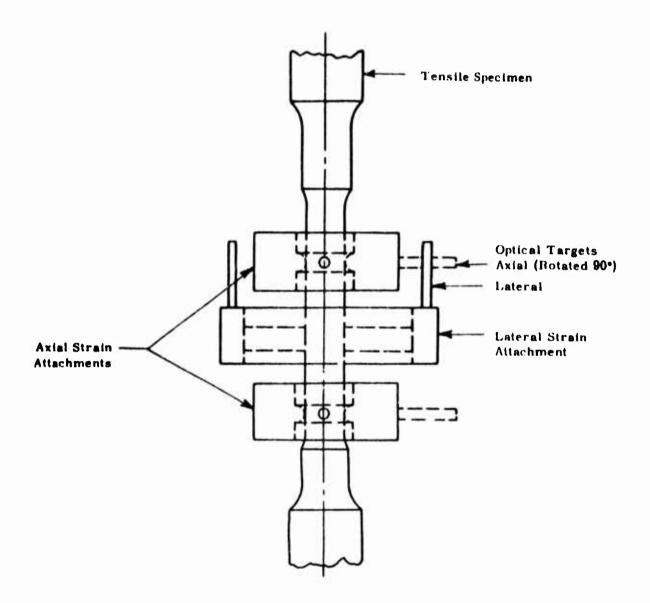


Figure 4. Location of the Flag Attachments on the Tensile Specimens

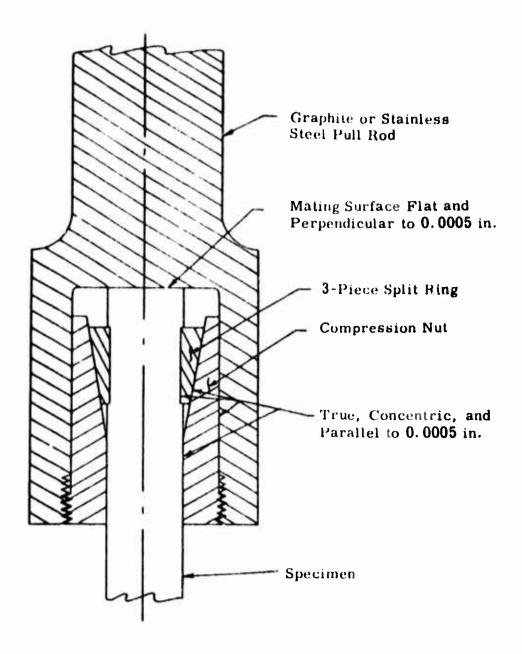


Figure 5. Precision Collet Grip for Tensile Specimen 2:1 Scale

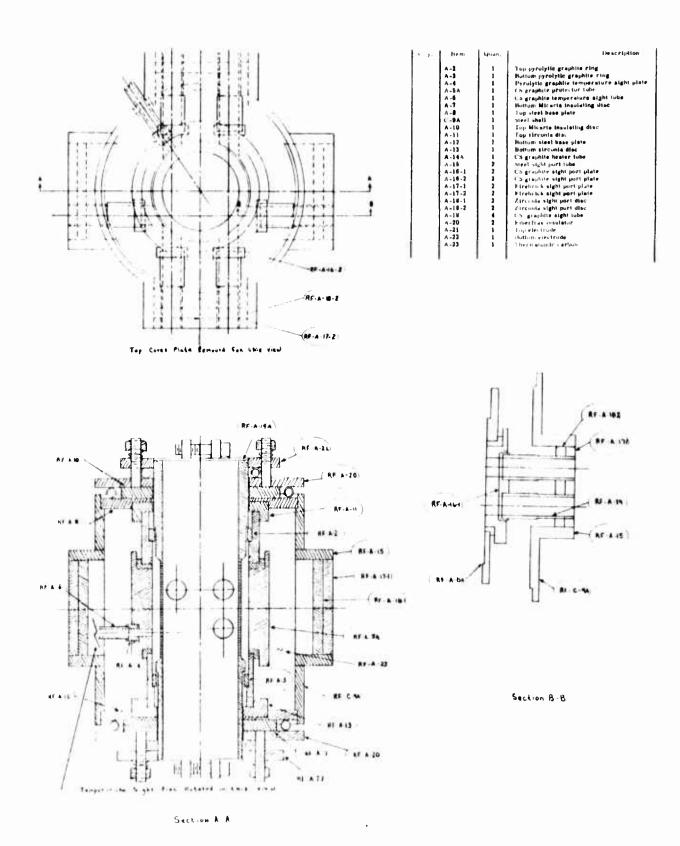


Figure 6. Small 5500°F Graphite Resistance Furnace

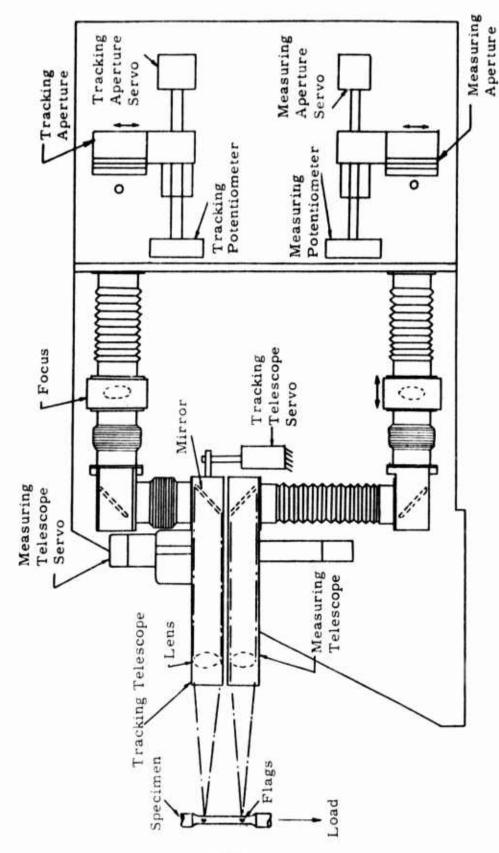


Figure 7. Arrangement of Optical Strain Analyzer

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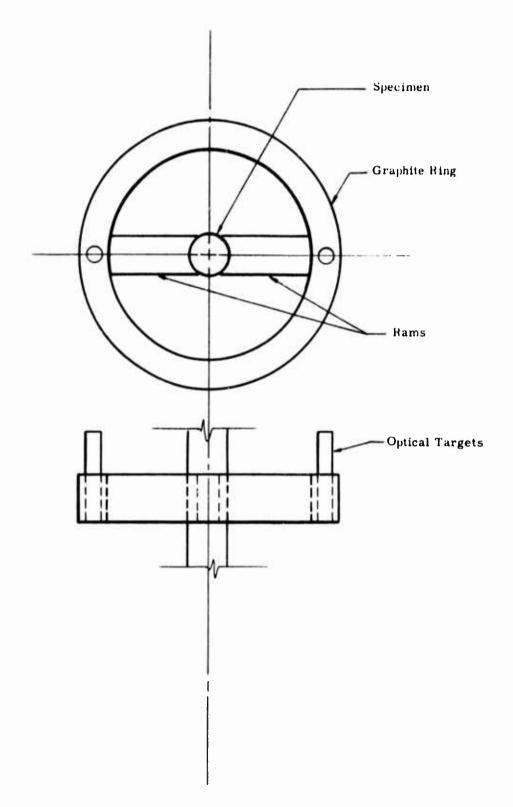


Figure 8. General Configuration of the Flag Attachment to Monitor Lateral Strain in Tension

APPENDIX B

ULTIMATE STRENGTH, ELASTIC MODULUS, AND POISSON'S RATIO TO 5500°F IN COMPRESSION

The compressive apparatus is shown in the photograph in Figure 1 and in the schematic in Figure 2 and consists primarily of a load frame, gas bearings, load train, 50-ton screw jack, variable speed mechanical drive system, strain analyzers, 5500°F furnace, and associated instrumentation for the measurement of load and strain.

The load frame is similar to most standard frames. It was designed to carry a maximum load of 100,000 pounds and to support the furnace optical strain analyzers, and other related equipment.

Gas bearings are installed at each end of the load train to permit precise alignment of the loading train to the specimen. The upper bearing is spherical on a radius of 6.5 inches. This radius is the distance from the top of the specimen to the spherical bearing surface. The load train, not the specimen, shifts to maintain radial alignment. The lower bearing is flat and is about 6 inches in diameter. The lower bearing permits transverse alignment of the load train. The gas bearings are floated for only a small initial amount of load so that precise alignment of the load train can be attained.

The load train near the furnace consists of the specimen loaded on each side by graphite and water-cooled steel push rods. The graphite push rods are counter-bored to permit insertion of a pyrolytic graphite disc which serves as a heat dam and to align the specimen to the center-line of the load train. Extreme care is exercised in the preparation of all parts of the load train to ensure concentricity of the mating parts to less than 0.0005 inch.

The 50-ton jack is a power screw type. The mechanical drive system consists of a gear reducer driven by a Louis Allis Synchro-Spede Unit (300-3000 rpm). The gear reducer is connected to the Synchro-Spede Unit through a chain coupling and to the 50-ton jack by a single roller chain and sprocket system. Different load rates are obtained by adjustment of the variable speed setting on the Synchro-Spede and by changeout of sprockets on the gear reducer and screw jack.

Figure 3 shows details of the "dumbbell" specimen which maintains a 0.500 inch diameter over the 1.2 inch long gage section. The specimen provides sufficient room for the flag attachments that follow the axial and lateral strains and also minimizes the influence of end restraint.

The flag attachments for the measurement of axial strain are positioned one inch apart so that unit strain is recorded directly. The flag attachment for the measurement of lateral strain is positioned between the flags for axial strain; see Figure 4. The lateral flag attachment used in compression is shown in Figure 5. The 4-piece assembly consists of a ring, two rams bearing on the specimen, and a screw to adjust the contact pressure. The ring was designed to track lateral motions as great as 0.030 inch without breaking.

Figure 6 is a sketch of the 5500°F furnace used for compression showing the basic components. The furnace consists of a resistivity heated graphite element insulated from a water-cooled shell by thermatomic carbon. The furnace and specimen are purged with helium to provide an inert atmosphere. Ports with visual openings are provided on opposite sides of the furnace as a means of allowing the strain analyzers to view the gage flags on the specimen. Specimen temperatures are determined by optical pyrometer readings taken through another small sight port containing a sapphire window. A calibration curve was established for the loss through the sapphire window, and since the furnace cavity acts essentially as a blackbody, true temperature readings are obtained. Power is supplied to the heating element by means of a 25 KVA variable transformer.

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upper target and the strain is monitored by the relative displacement of the aperture rather than the telescope following the lower target. With this procedure the maximum range is the maximum displacement available for the lower aperture, or about 1/8 inch, and the sensitivity is limited by the optics and the noise level of the detector. Using both telescopes, the range is about 3/4 inch.

To provide optical references on the specimens, targets are affixed to the test specimen as mentioned. When the specimen is heated to temperature, the targets are self-luminous and are observed optically. The optics view past the luminous targets into a cooled cavity in the opposite furnace wall. The self-luminous targets are then visible against a dark background. To obtain data at below 2000°F, a light beam is directed from behind the flags providing a shadow image for the detection system.

The image of the glowing target is focused through a rotating shutter (chopper) and onto a rectangular aperture. Small slits in the aperture pass a portion of the upper and lower edges of the light beam. A photocell receives the light thus transmitted, and an electronic circuit detects whether the energy passed by the two slits is equal. A servo drives the apertures to let a balanced quantity of light pass through the two slits and thus maintains an optical null.

To obtain lateral strain, a strain analyzer is supported horizontally on the load frame to view the diametrical or lateral strain of the specimen.

Calibrations of the analyzers are performed in various ways including absolute correlations to precision micrometers, strain gage extensometers, and direct plots of stress-strain for reference materials such as steel, plexiglas, magnesium, and aluminum. Precision is ± 0.000020 inch.

Instrumentation includes primarily a stress-strain measurement system composed of a 20,000-pound SR-4 Baldwin load cell, constant d.c. voltage power supply, two optical strain analyzers, and two X-Y recorders. Specimen temperature is monitored with an optical pyrometer. Stress(load) is measured by a commercial load cell. The cell receives a constant d.c. voltage input from the power supply and transmits a millivolt signal (directly proportional to load) to an X-Y recorder. Simultaneously, the optical strain analyzers measure both the axial and lateral strain and transmit a millivolt signal (proportional to strain) to the X-Y recorders. Thus, continuous plots of stress-axial strain and axial strain-lateral strain are recorded simultaneously.

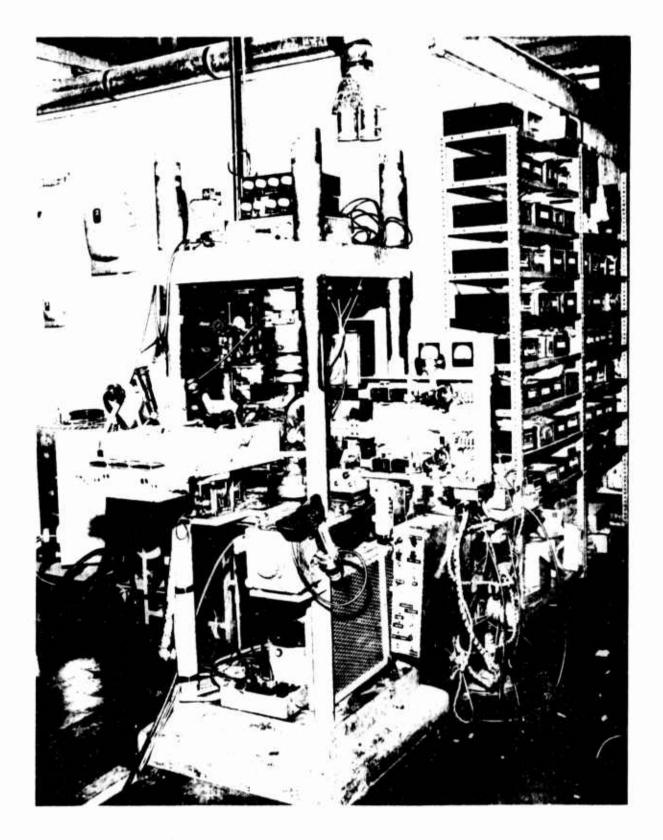


Figure 1. Picture of the Compressive Facility with Gas Bearings and Optical Strain Analyzer

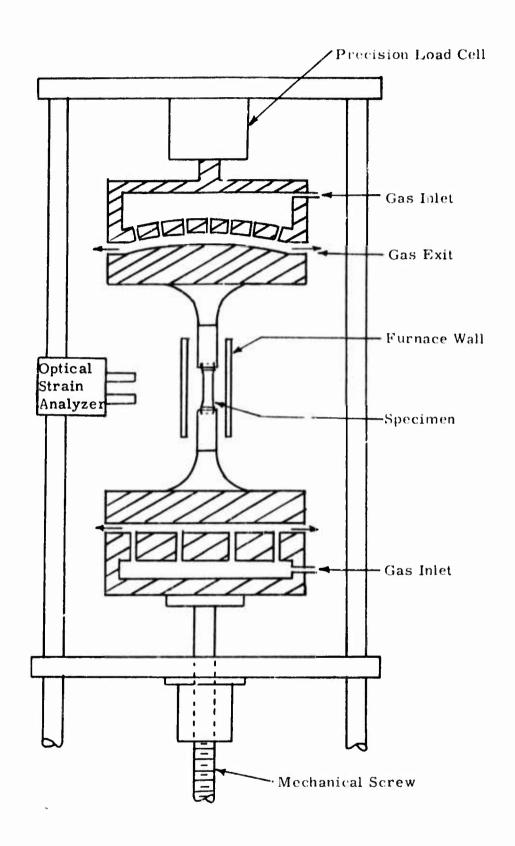
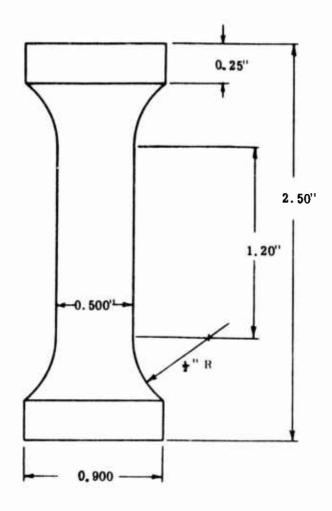


Figure 2. Schematic Arrangement of Gas-Bearing Universals, Specimen, and Load Train



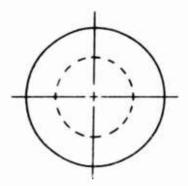


Figure 3. Compressive Specimen Configuration

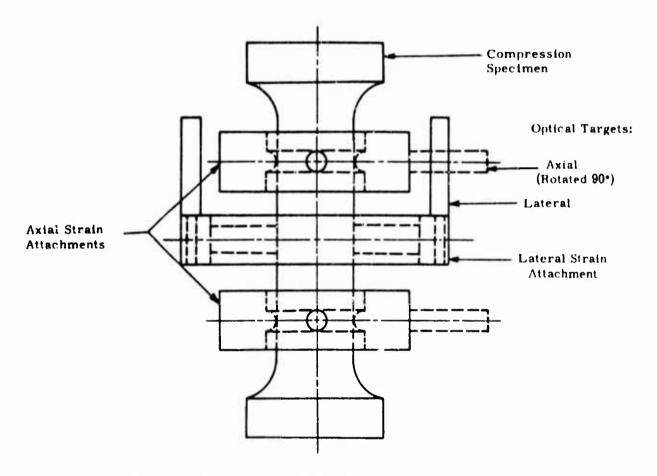


Figure 4. Location of the Flag Attachments on the Compressive Specimen

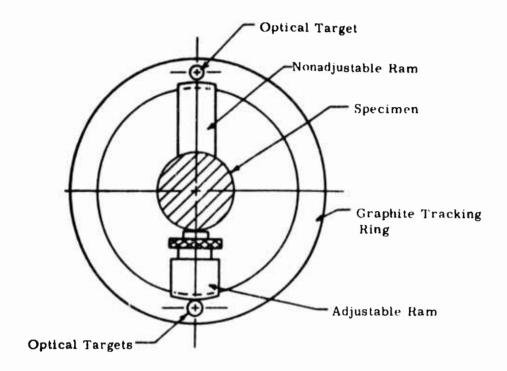


Figure 5. Lateral Strain Flag Attachment for Compressive Specimen

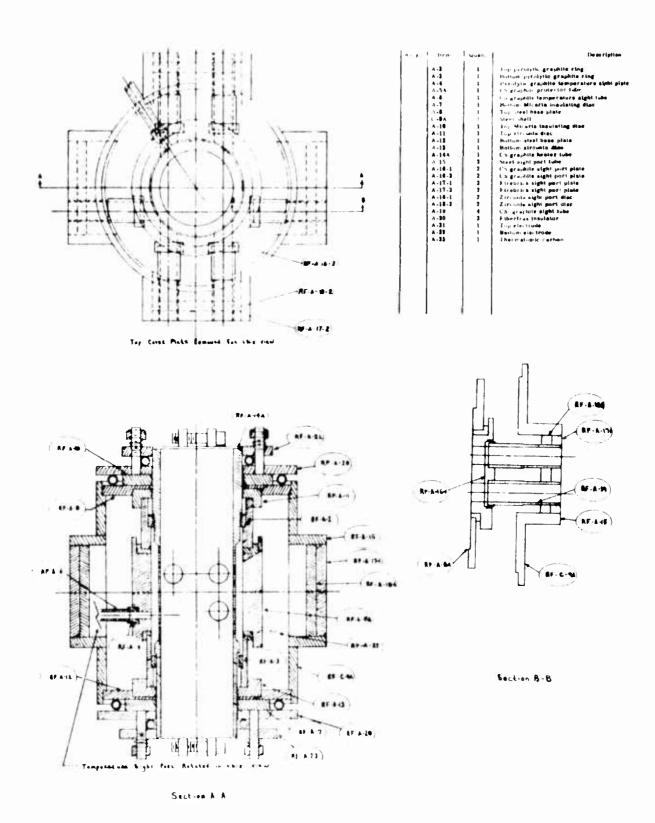


Figure o. Small $5500^{\circ}\mathrm{F}$ Graphite Resistance Furnace

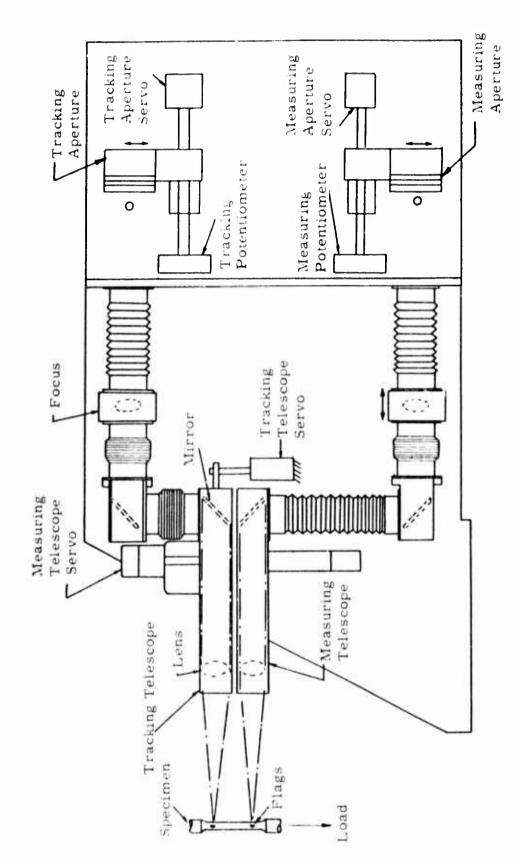
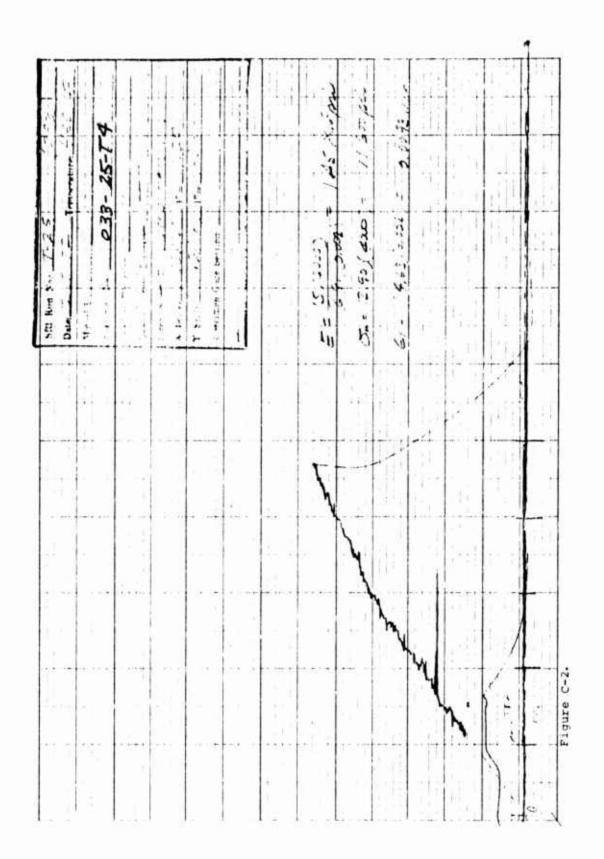


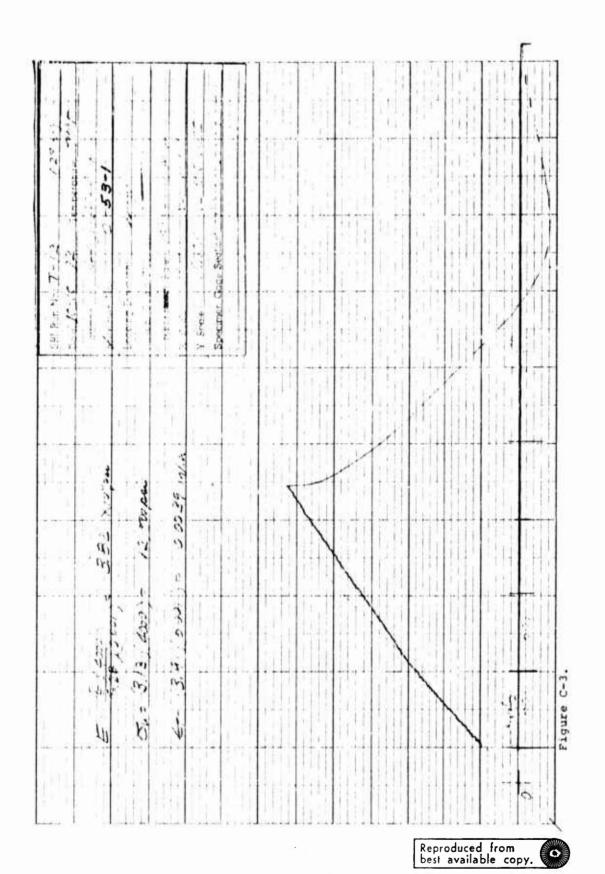
Figure 7. Arrangement of Optical Strain Analyzer

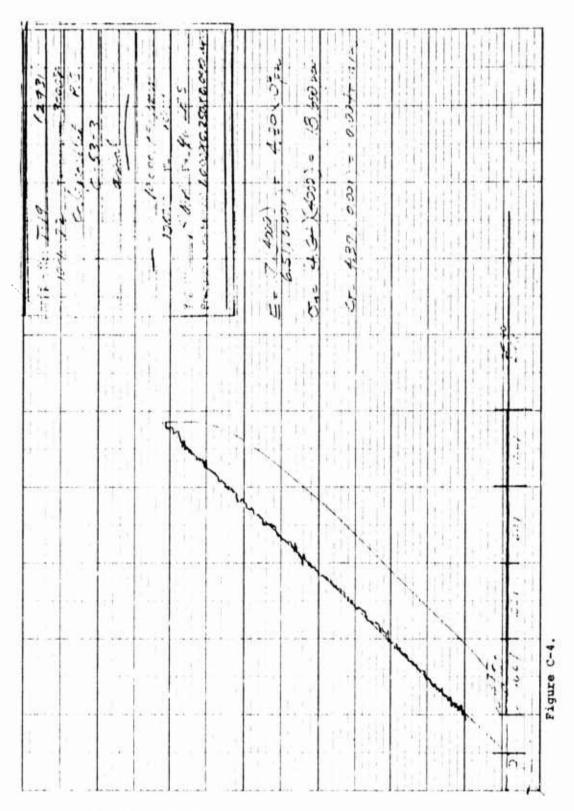
APPENDIX C

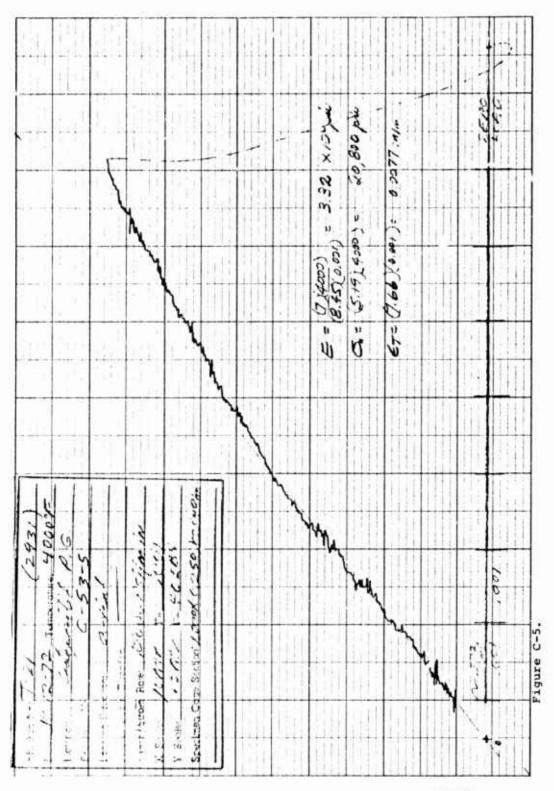
TENSILE STRESS-STRAIN CURVES FOR PG AND CODEPOSITED SIC/PG IN THE AB DIRECTION

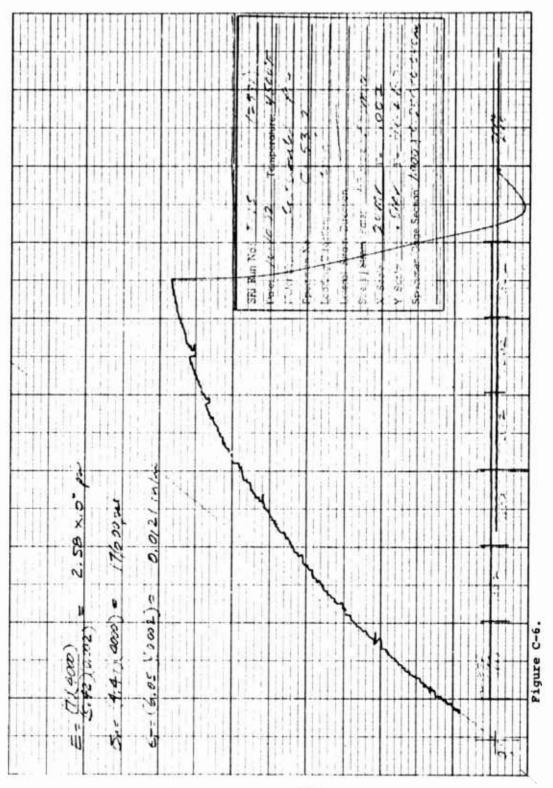
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			Figure C-1.
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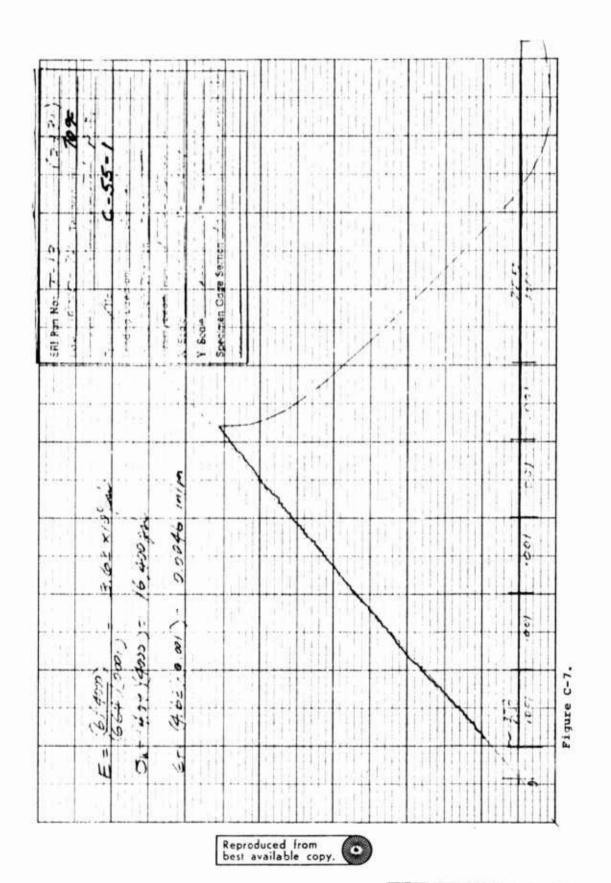


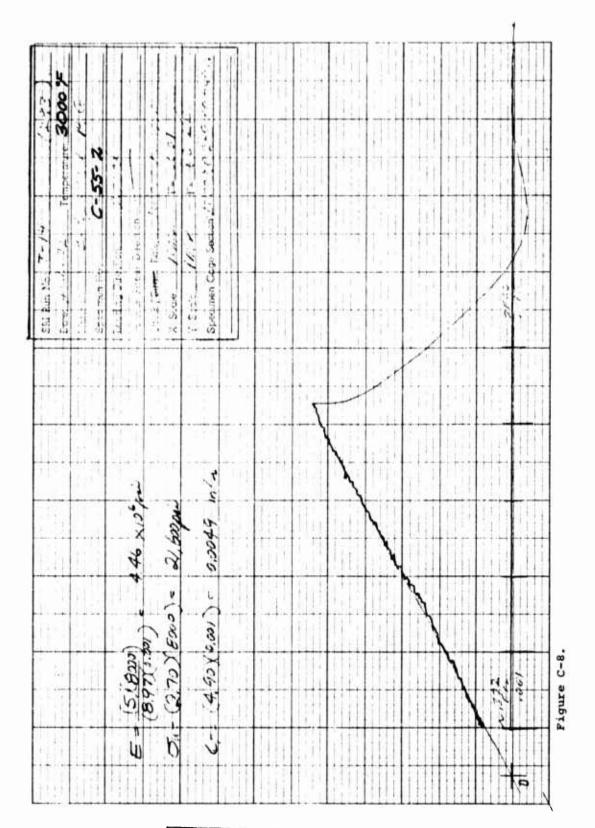


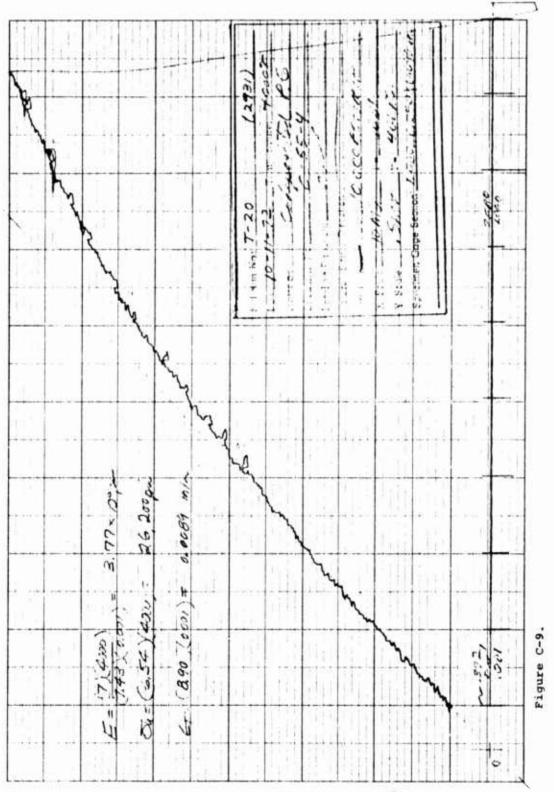


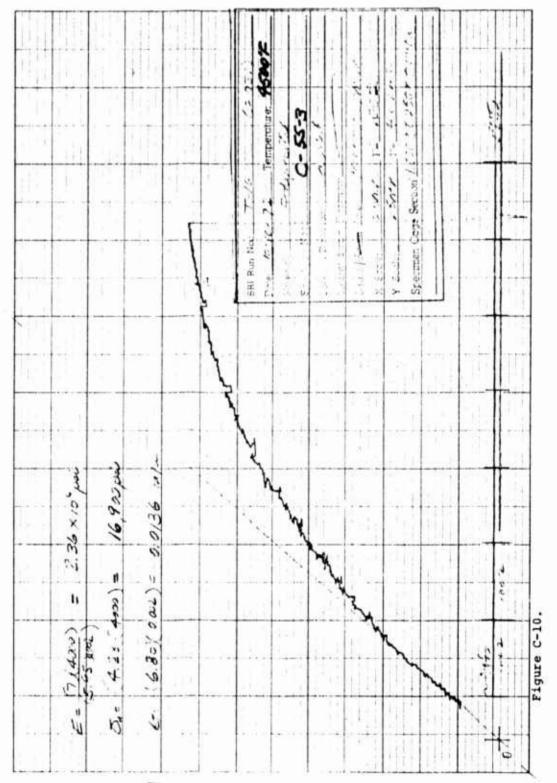


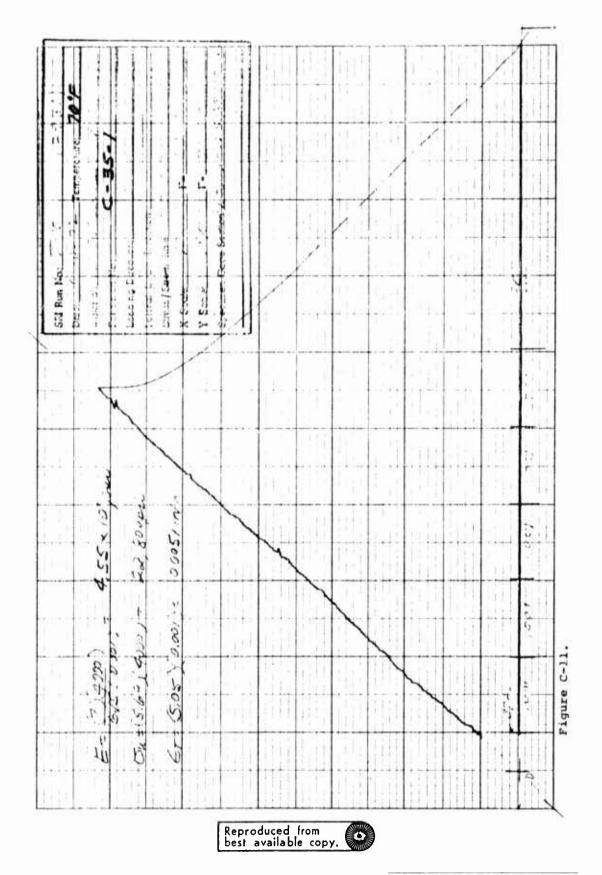
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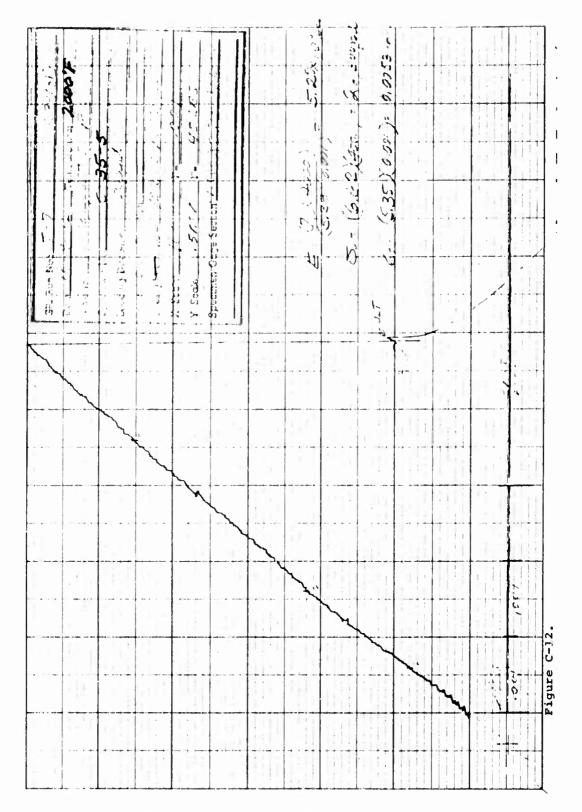




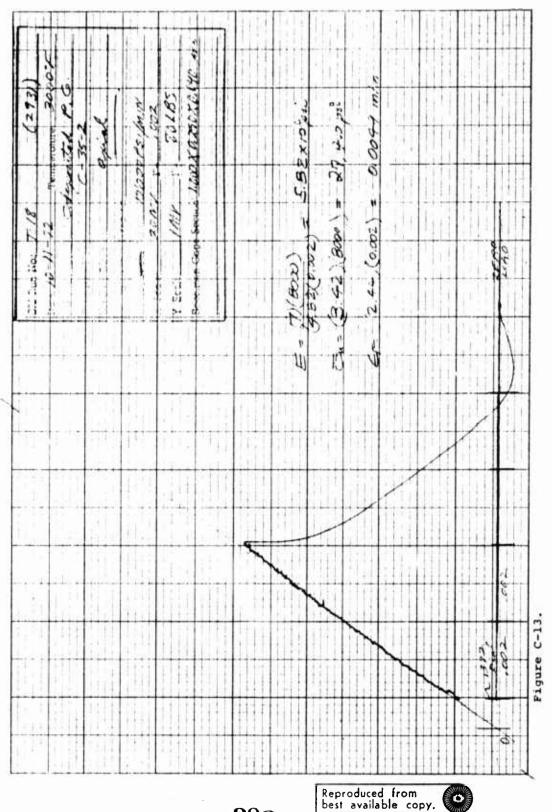




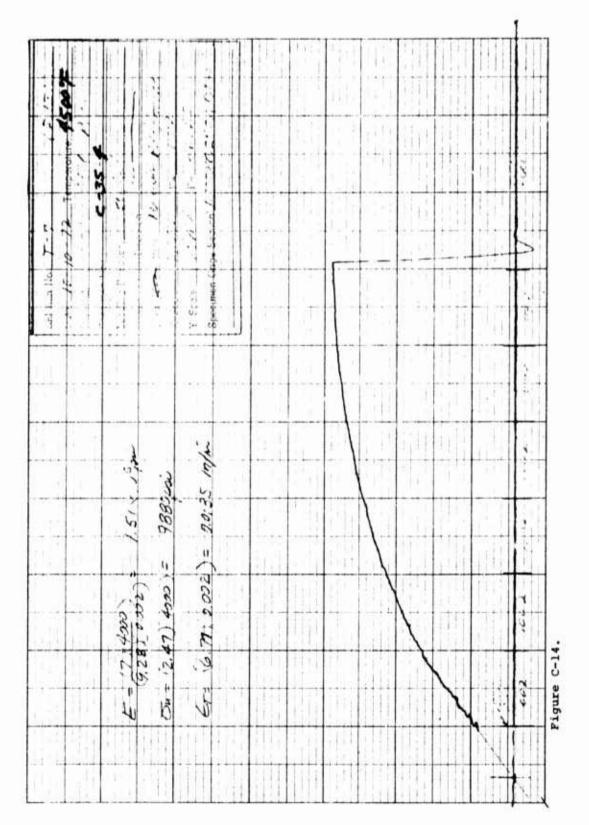




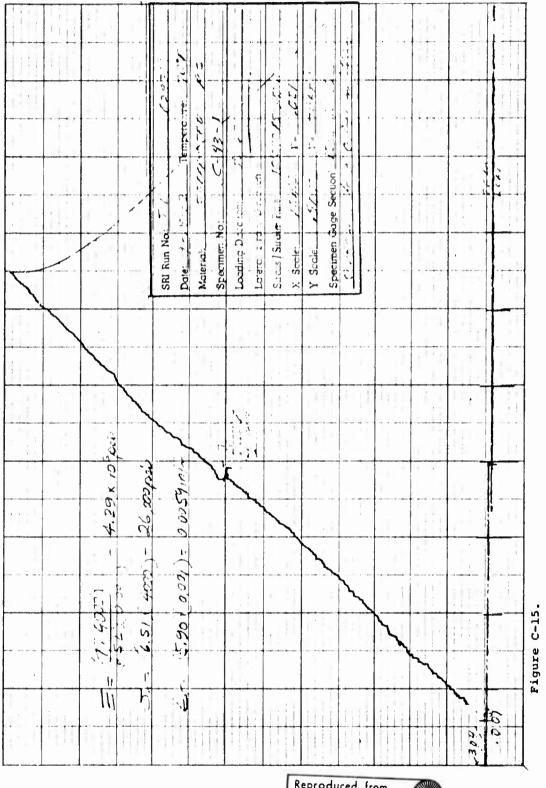
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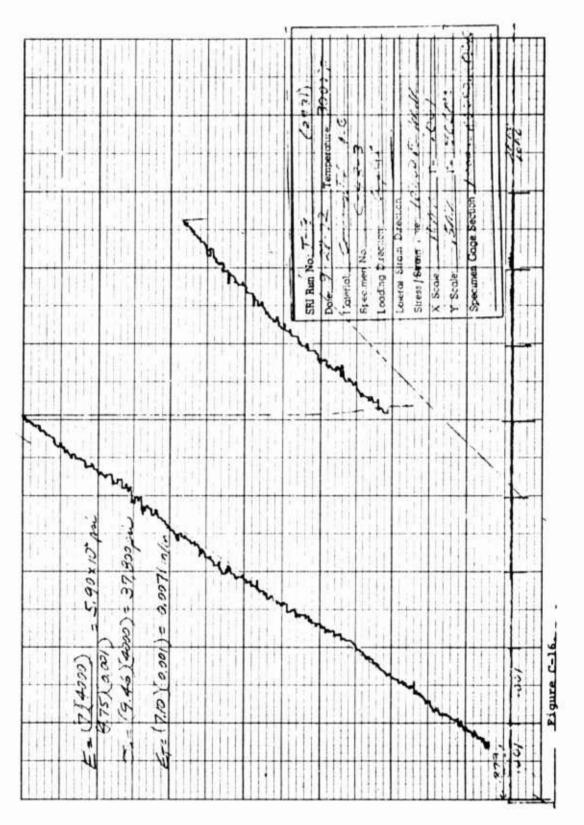


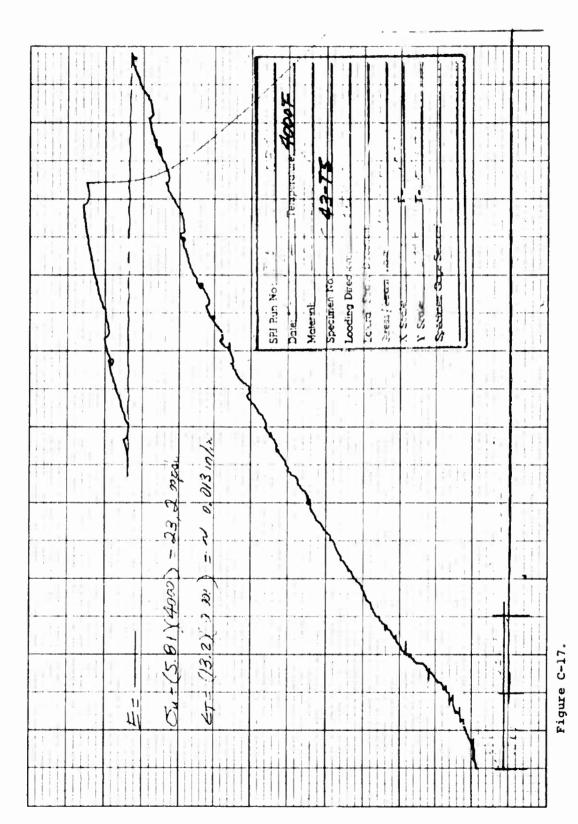
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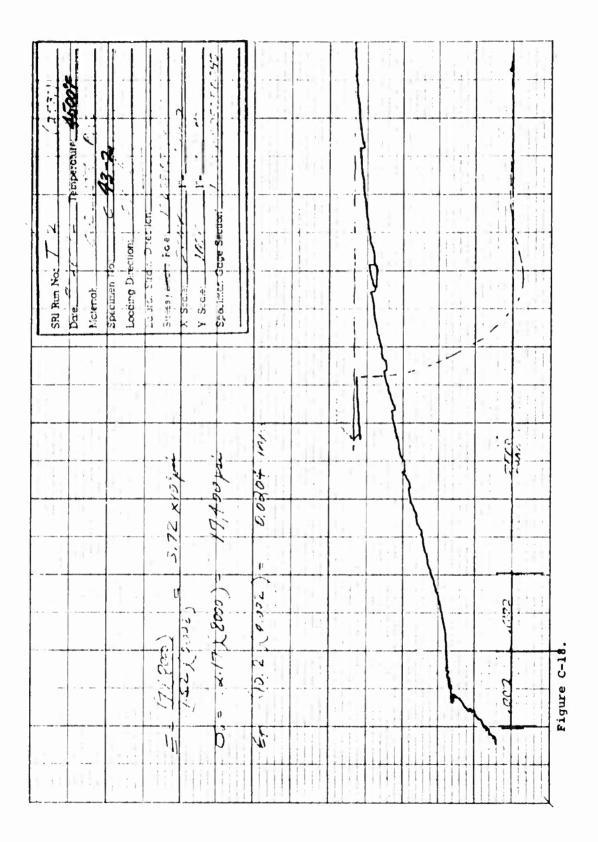
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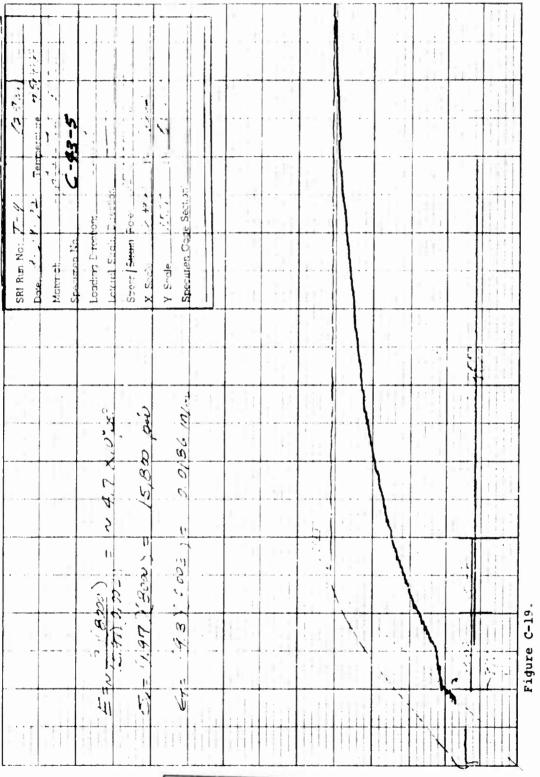




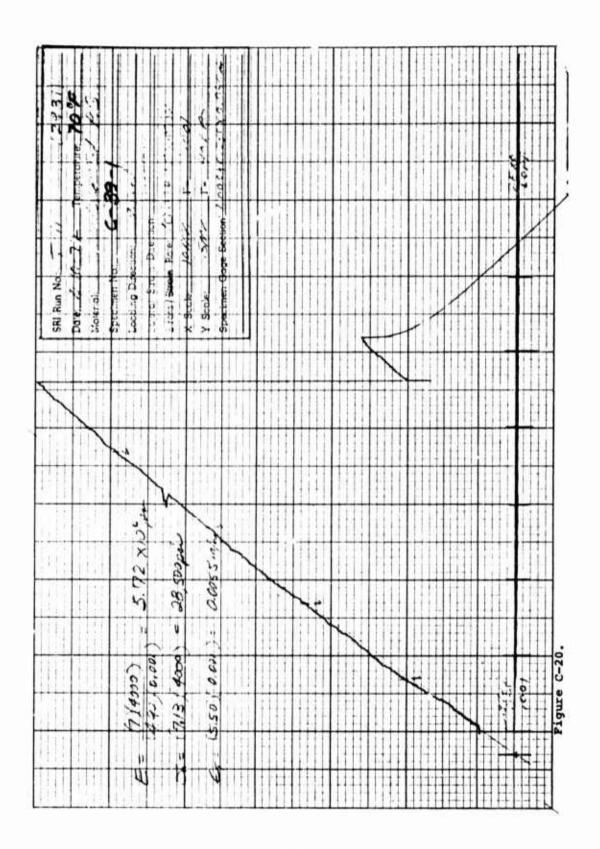
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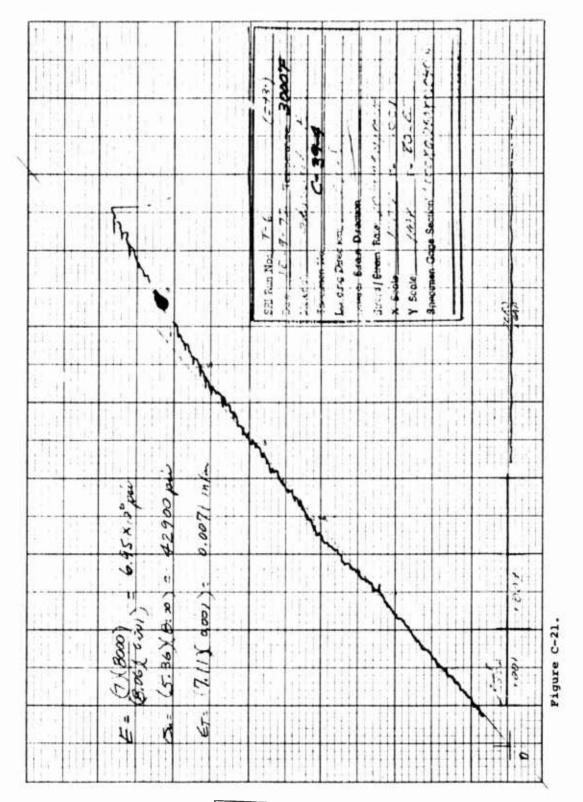


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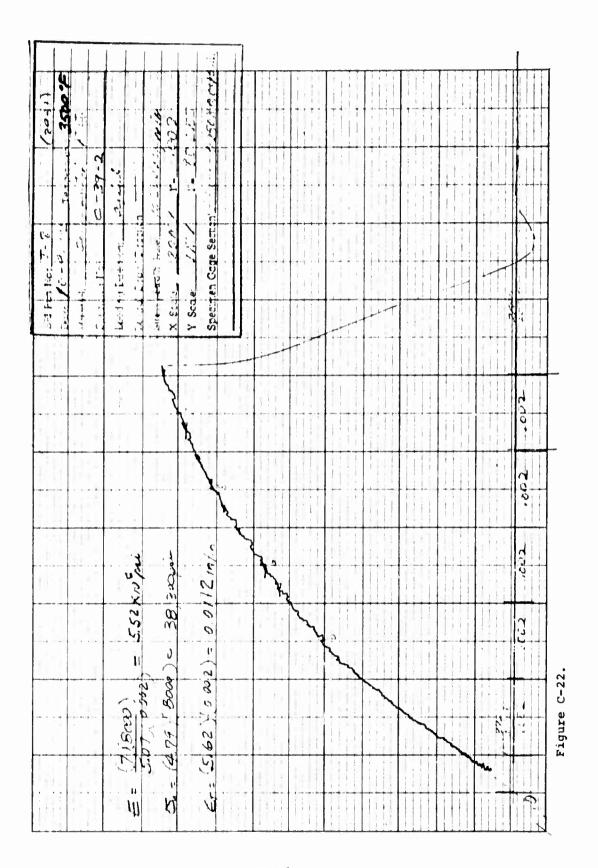


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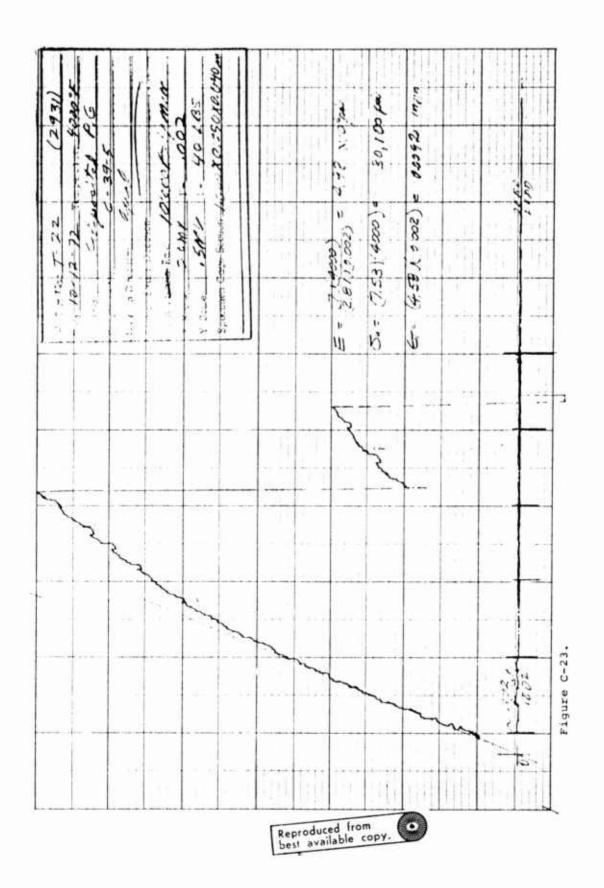


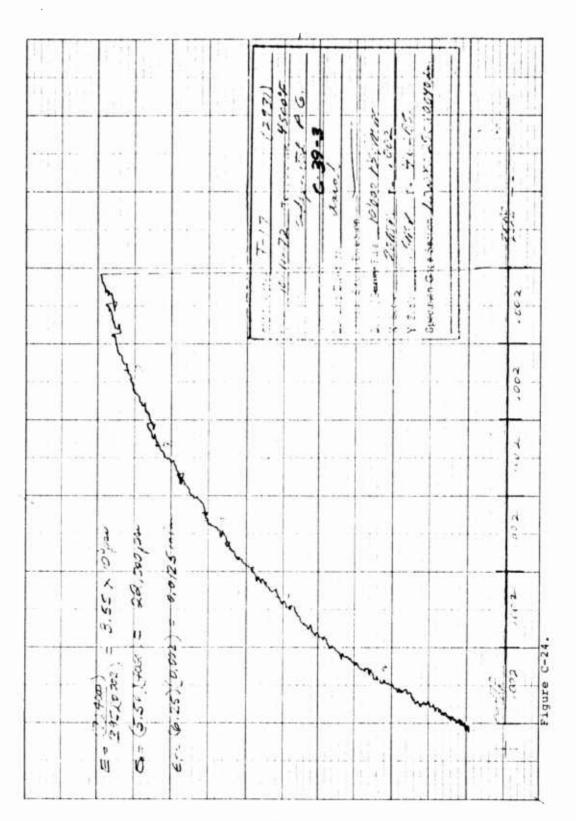


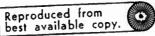
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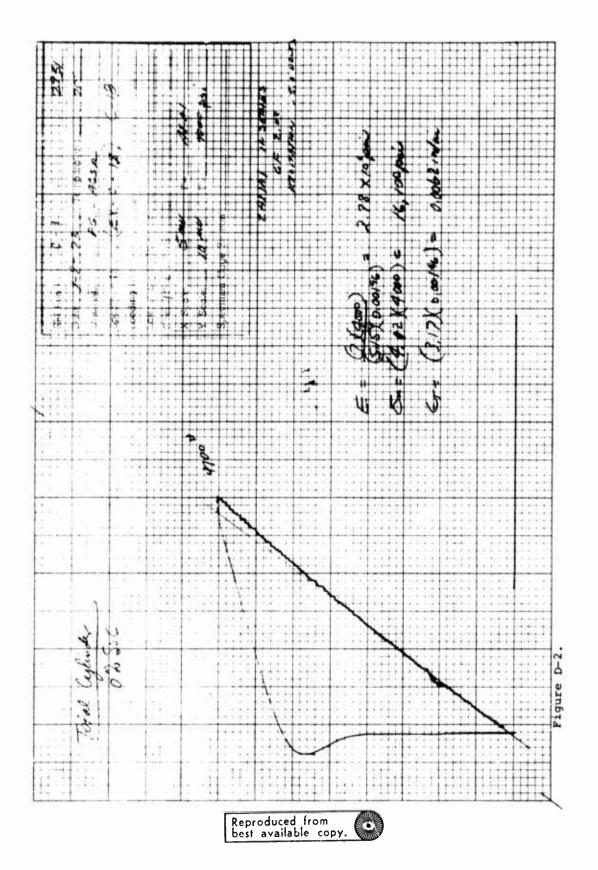


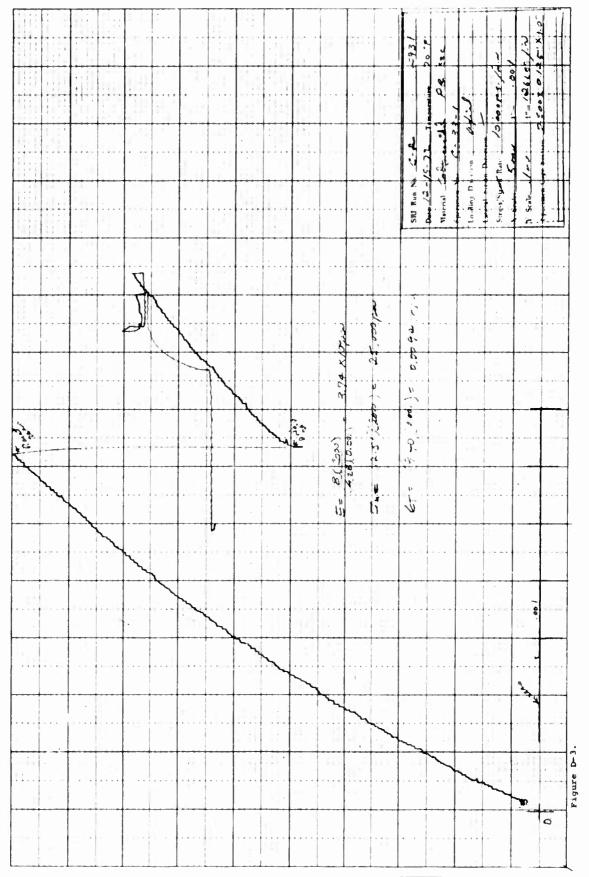


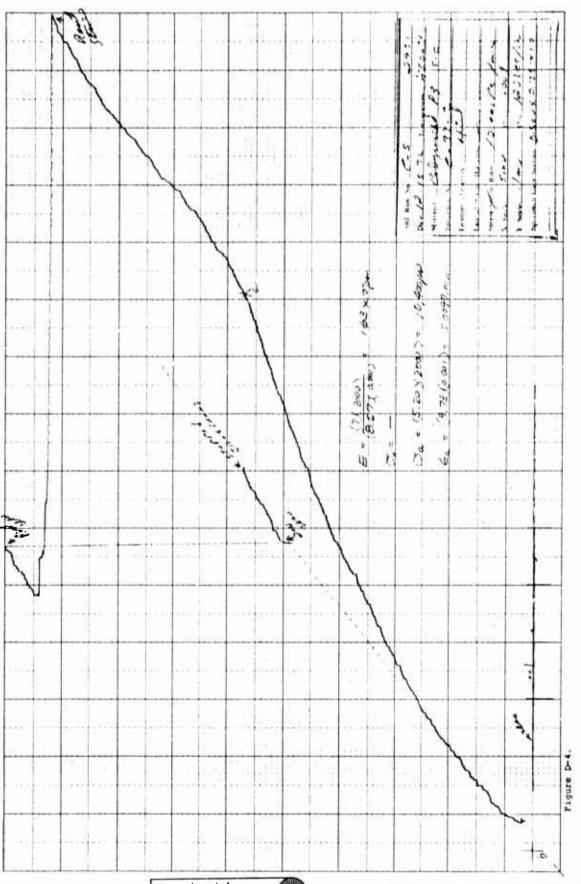
APPENDIX D

COMPRESSIVE STRESS-STRAIN CURVES FOR PG AND CODEPOSITED SIC/PG IN THE AB DIRECTION

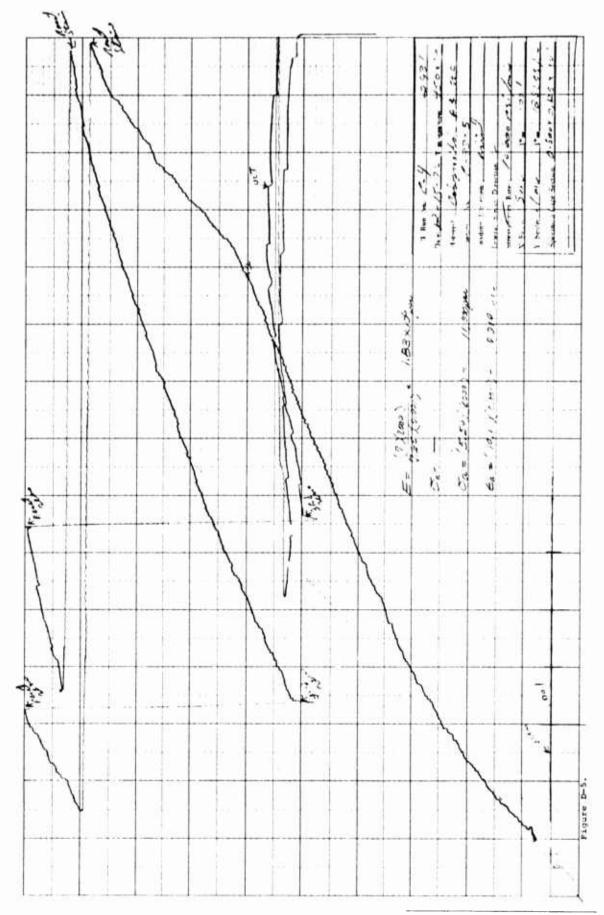
215

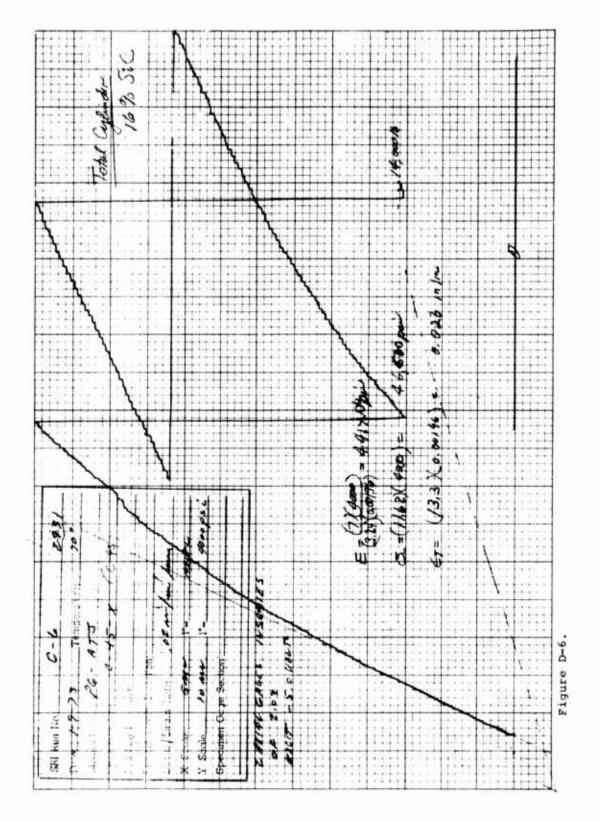


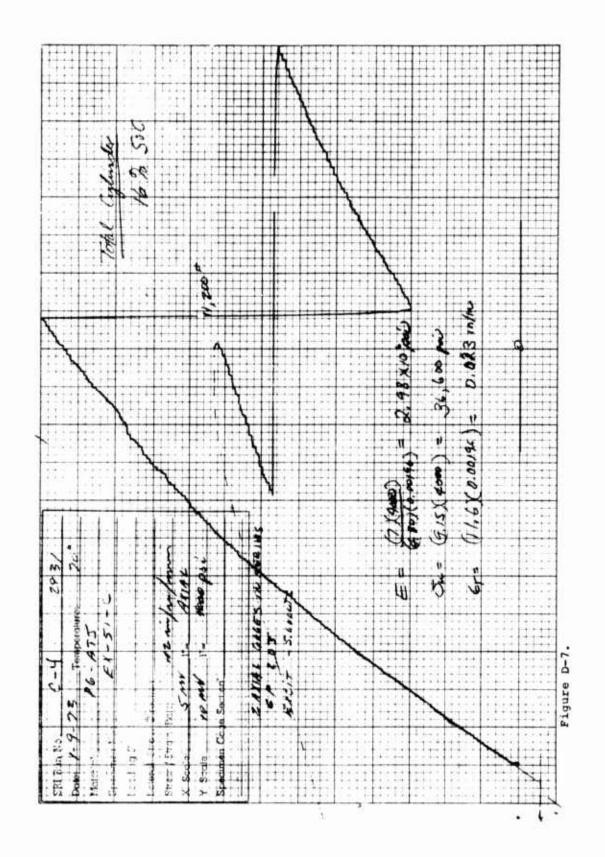


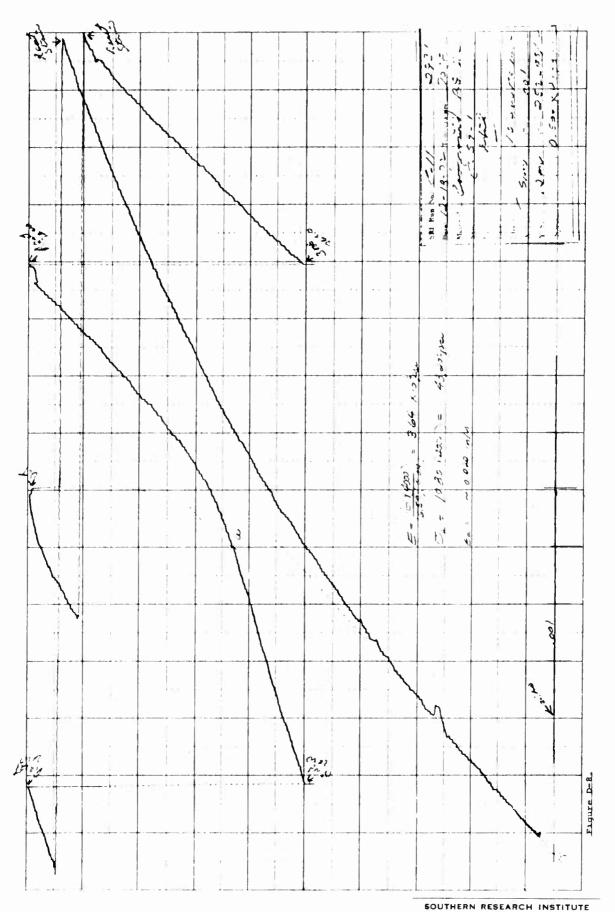


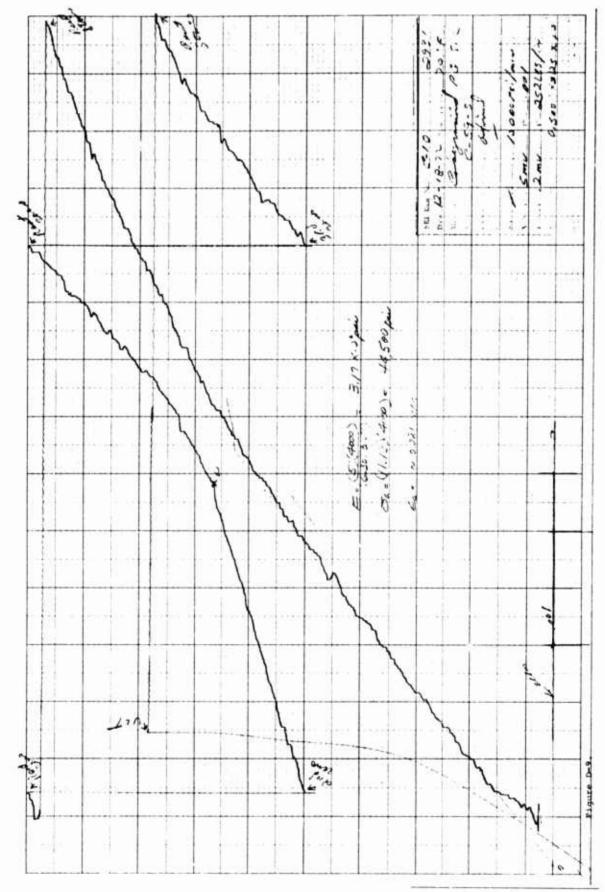
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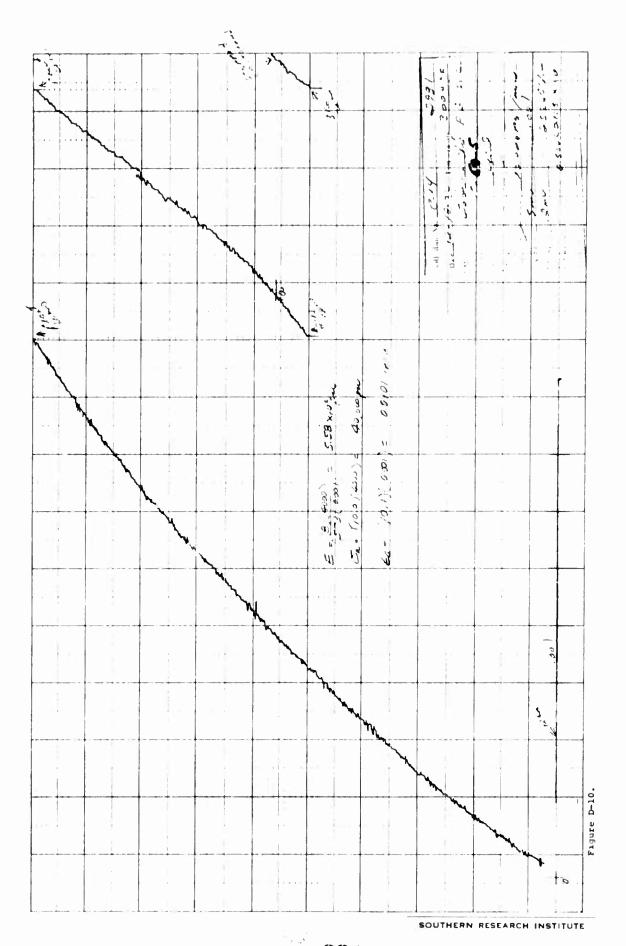


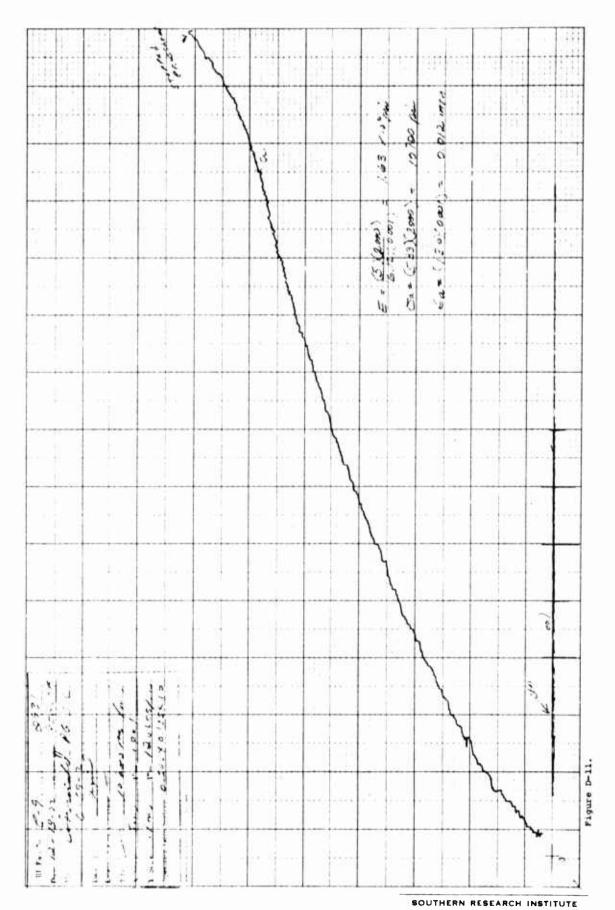


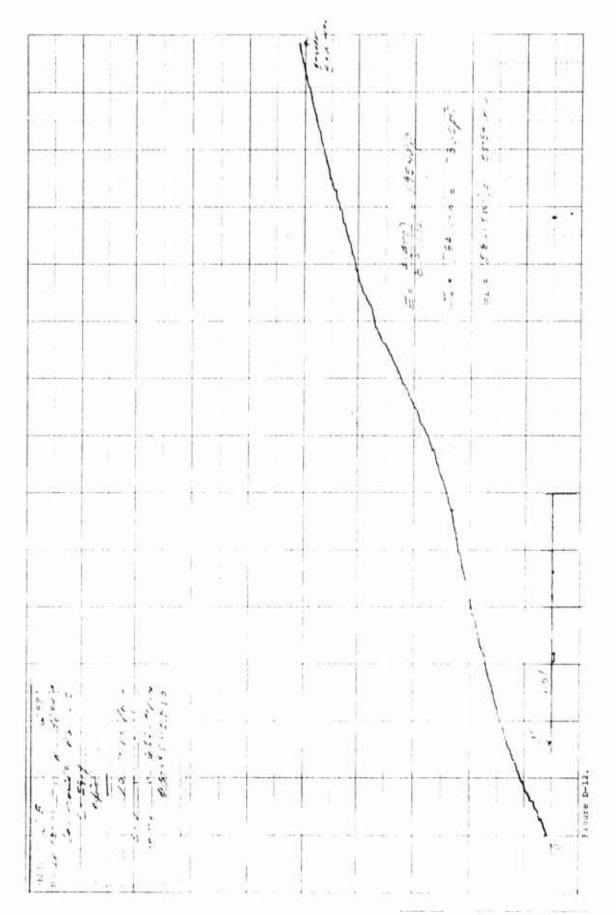


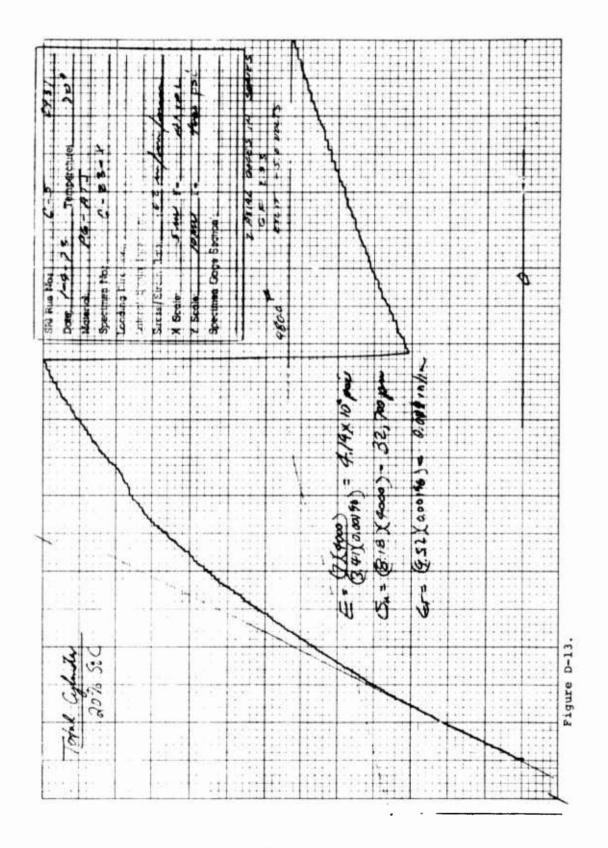




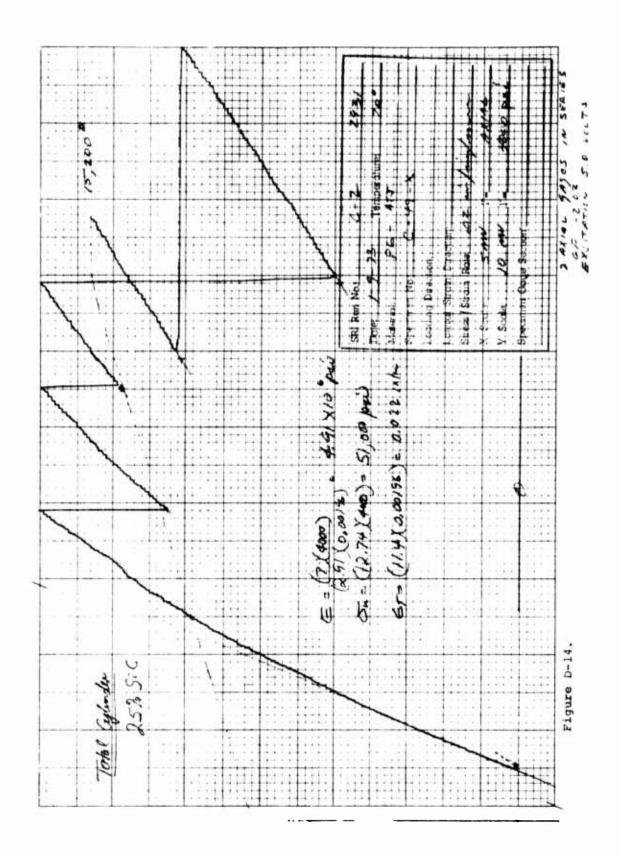


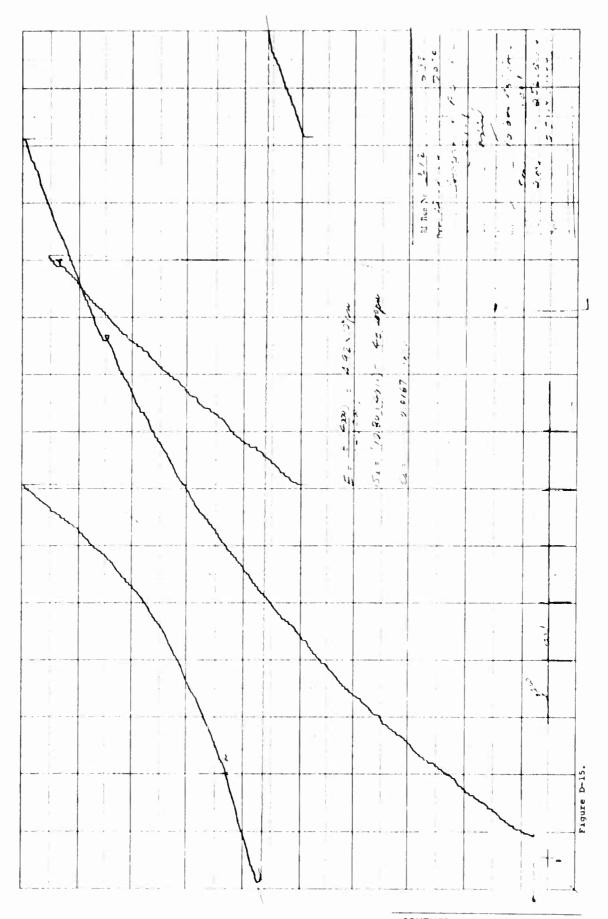


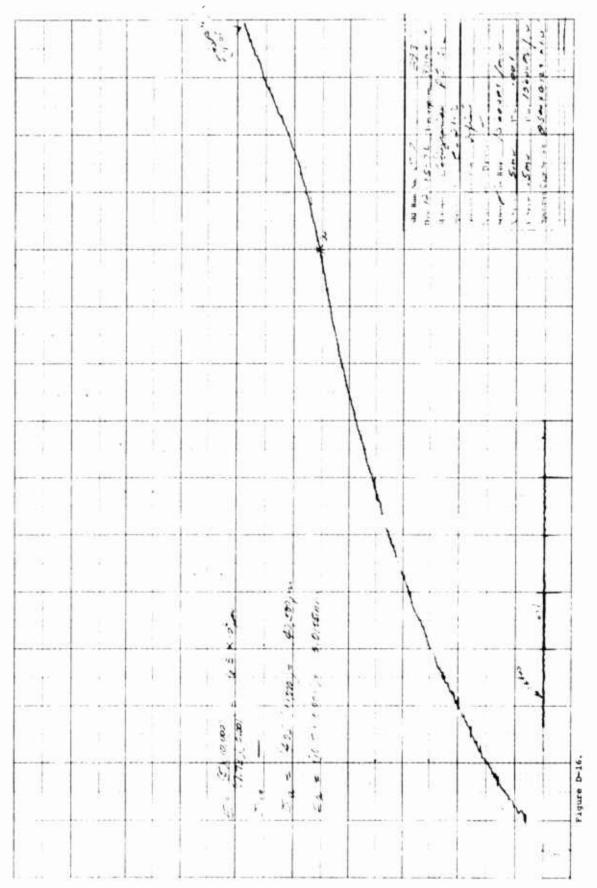


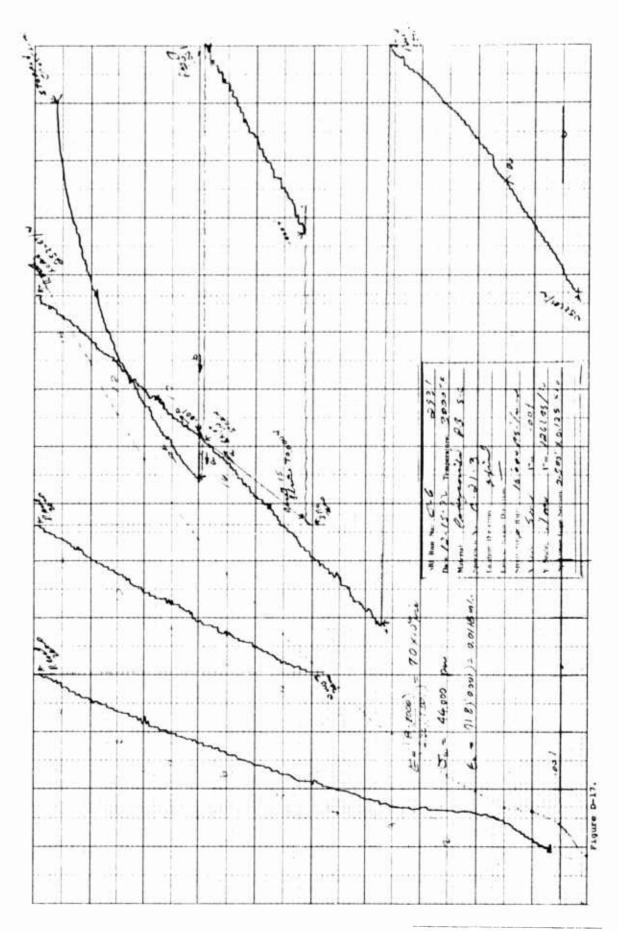


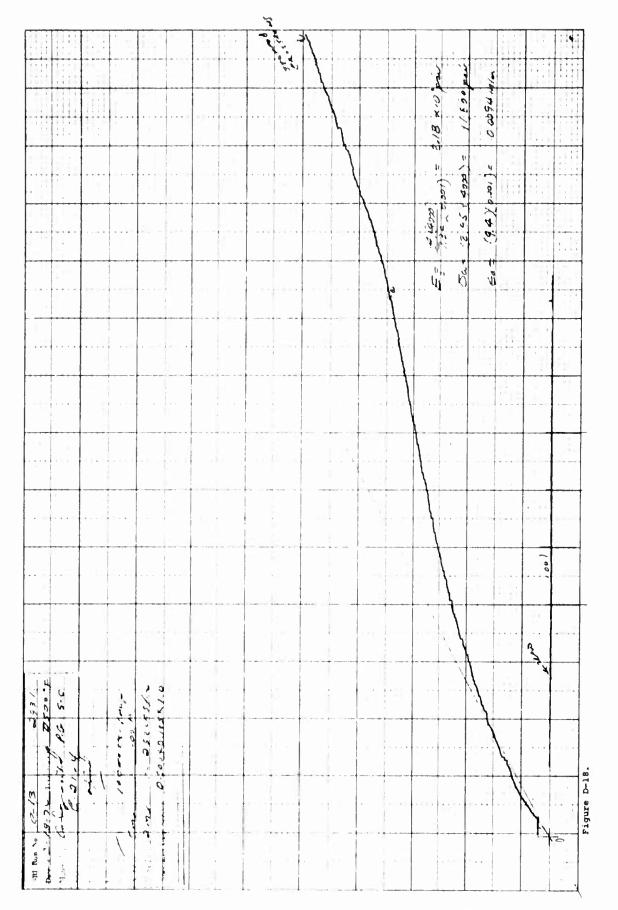
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APPENDIX III

FLEXURAL STRENGTH AND ELASTIC MODULUS OF PG/SiC

111-1

Flexural strengths and elastic moduli were determined at room temperature using 1-inch radius cylindrical and plate stock that had been fabricated for the SoRI thermal property characterization program. The third cylindrical exit piece for ab direction thermal properties determination at SoRI and 1 of the 4 plate pieces for c directionthermal properties at SoRI were selected to give a range of SiC contents. Are segments taken from rings which were in turn taken from the cylinders were broken with the substrate side in tension in the specially constructed fixture shown in Figures III-1 and III-2. Flat plate specimens were similarly broken. The fixture consists of an aluminum cylindrical shell with a steel base that fits inside of it. The steel base has two parallel 0.125-inch radius steel pins 1-inch apart built into it. The third steel pin is built into a steel plunger as shown. Specimens are then broken in 3-point loadings. A steel ball transmits the load from the cross-head platen to the plunger. The ball ensures good alignment provided the sides of the specimen are square and parallel. The fixture eliminates the need for perfectly aligned stationary and movable fixturing. The specimen is simply placed in the fixture which is then placed on the anvil of the tensile machine and the cross-head is moved downward at a rate of 0.2 in/min. The load and the deflection are plotted automatically on a strip-chart recorder. A model Tinius-Olsen tensile machine was used for this testing. The broken specimens were assayed for SiC content by ashing.

The following formulae were used to calculate the flexural strengths and elastic moduli:

$$S = \frac{1.5\ell}{bh^2}$$

and

$$E = \frac{P\ell^3}{4ybt^3}$$

where

S = strength, psi

E = elastic modulus, psi

P = loads, lb

Q = span length, in. = 1

b = specimen width, in.

h = specimen height, in.

t = deflection, in.

Use of the elastic modulus was based on the elastic modulus being the same in tension as it is compression. This turned out to be a reasonably good assumption as the data in Tables 2 and 4 of Appendix II show. A small



Figure III-1. Flexural Test Fixture.

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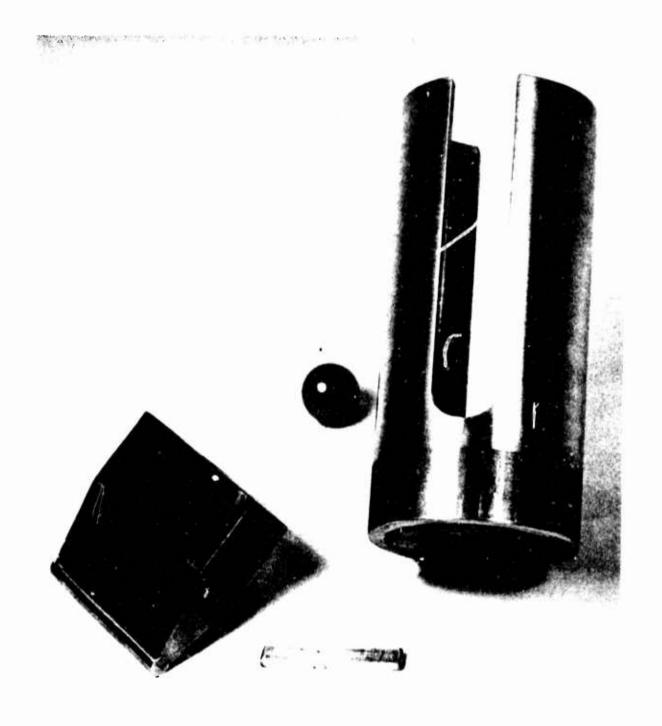


Figure III-2. Flexural Test Fixture Exploded. ${\bf 236}$

correction was applied for the 1-inch radius are specimens. This correction was a function of radius, r, to thickness, t, ratio as follows:

r/t	Percent Error (S)	Percent Error (E)		
1	35.0			
2	17.0			
3	10.9			
4	9.2			
10	3.2			

The r/t for these specimens was on the order of 10. The results are shown in Table III-1.

The height measurement, h, was made on the fractured specimen, at the break, using an optical method. The minimum height was used for these specimens, which, by their nature, are somewhat rough, as deposited.

The microstructure was mostly Code 20 which contains a fine even dispersion of SiC, with diameters ranging from 0.9 to 2.6μ and and L/D of 3 or more, in a strain-line-free pyrographite matrix. The needles coarsened and tended to segregate in the 12 C grain boundaries at the higher SiC concentrations. It is suspected that the SiC contents may be somewhat on the low side, particularly at the higher concentrations, since the ashing technique at Atlantic Research has been shown to be somewhat in error.

Table III-1. Flexural Strength Data — PG/SiC, "ab" Direction.

LOG RUN NO.	% SiC BY ASHING	_b	<u>h</u>	P	t	FLEXURAL STRENGTH (Ksi)	MODULUS OF ELASTICITY (Msi)
CYLINDERS							
162-32 ENTR.	13.0	0.255	0.150	133.5	_	37.0	-
162-33 EXIT	11.8	0.244 0.248	0.145 0.165	116.5 90.3	0.0110 0. 008 0	35.7 21.3	3.61 2.52
162-45 EXIT B	21.3	0.247 0.247	0.135 0.130	102.9 95.0	0.0080 0.0075	35.7 35.7	5.30 5.85
162-7 EXIT	19.0	0.265	0.075	3C 2	_	31.2	_
162-11 ENTR.	24.2	0.240	0.135	103.7	_	37.1	_
162-39 EXIT	18.4	0.237 0.237	0.105 0.110	51.1 51.1	0.0105 0.0095	30.5 27.7	4.45 4.25
162-44 ENTR.	31.5	0.251	0.145	104.0	-	31.1	_
162-12 EXIT	34.7	0.255 0.240	0.060 0.060	23.5 22.0	0.0155 0.0140	39.5 39.2	6.85 7.45
162-39 ENTR.	27.8	0.250	0.150	106.0	_	29.7	_
162-12 ENTR.	27.4	0.240	0.085	35.4	_	30.6	_
162.44 EXIT A	42.5	0.245 0.245	0.100 0.100	62.7 59.7	0.0072 0.0070	39.7 40.1	8.82 8.73
PLATE							
162-26	19.2					28.1	-
162-28	16.9					34.0	_
162-35	11.6					23.1	-